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PATENT ABSTRACTS OF JAPAN

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(54) COMBINED HARD COATING MEMBER

(57)Abstract

PROBLEM TO BE SOLVED: To solve the problem that a conventional combined hard coating member composed of a Ti-Al-containing compound is inferior, because of many defects and strain of the coating itself, in the adhesion between the coating and a material adjacent to it and is liable to suffer peeling of the coating and is also inferior in the various properties of the coating itself and, for example, in the case of use as a cutting tool, this member rapidly causes the development of peeling, fine chipping, wear, or the like, due to the defects and strain of the coating itself and has only a short life.

SOLUTION: In the member coated with a combined hard coating, the surface of a base material is coated with a single-layer or a laminated coating layer including a combined hard coating of at least one kind among titanium- and aluminum-containing compound nitrides, compound carbides, compound carbonitrides, compound nitroxides, compound carboxides and compound carbonitroxides. Moreover, when X-ray diffraction is performed using a copper target on the surface of this coating, the following inequalities are satisfied: $h(200)/h(111) \geq 4.0$, where $h(200)$ and $h(111)$ represent the peak heights of the (200) crystal plane and the (111) crystal plane, respectively; and $1.5 \leq d(200)/d(111) \leq 0.8$, where $d(200)$ represents the half-width of the peak of the (200) crystal plane and $d(111)$ represents the half-width of the peak of the (111) crystal plane.

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CLAIMS

[Claim(s)]

[Claim 1] The compound nitride, compound carbide which contain titanium and aluminum on the surface of a base material, When it is covered as an enveloping layer of the monolayer containing at least one sort of compound hard layers in a compound charcoal nitride, a compound nitric oxide, a compound carbonation object, and a compound charcoal nitric oxide, or a laminating and X-ray diffraction is carried out from the front face of this compound hard layer using a copper target (200) When the peak height of the crystal face is set to h (200) and the peak height of the crystal face (111) is set to h (111) The compound hard layer covering member which consists of $h(200) / h(111) \geq 4.0$, and consists of $1.5 \geq d(200) / d(111) \geq 0.8$ when the half peak width of the peak of the ** (200) crystal face is set to d (200) and the half peak width of the peak of the ** (111) crystal face is set to d (111).

[Claim 2] The above-mentioned base material is a compound hard layer covering member according to claim 1 which consists of a cemented carbide containing at least one sort as which 4 - 15 % of the weight and the remainder were chosen out of 4a and 5a of a tungsten carbide or a tungsten carbide, and a periodic table, the carbide of 6a group metal, charcoal nitrides, and these mutual solid solutions in the joint phase which a principal component becomes with Co and/or nickel of hard phases.

[Claim 3] The above-mentioned base material is a compound hard layer covering member according to claim 1 to which the remainder becomes 80 - 98 % of the weight from the silicon-nitride system sintered compact which is a sintering acid about a silicon nitride and/or sialon.

[Claim 4] The above-mentioned base material is a compound hard layer covering member according to claim 1 which consists of a super-elevated-temperature hyperbaric-pressure sintered compact containing at least one sort of grain-boundary joint phases as which 20 - 90 % of the weight and the remainder were chosen in the cubic boron nitride from the nitride of Ti, aluminum, Mg, and Si, borides, and these mutual solid solutions.

[Claim 5] The above-mentioned base material is a compound hard layer covering member given in any 1 term of the claims 1-4 which the surface roughness of this base material becomes from 0.1 micrometers or less by the mean surface roughness by Ra of JIS.

[Claim 6] The above-mentioned enveloping layer is a compound hard layer covering member given in any 1 term of claims 1-5 which becomes by the above-mentioned compound hard layer.

[Claim 7] The above-mentioned compound hard layer is a compound hard layer covering member given in any 1 term of claims 1-6 which consists of the cubic type crystal structure which layer thickness becomes by 1-15 micrometers.

[Claim 8] the above-mentioned compound hard layer -- w(Cx (Tia, Alb), Ny, Oz) [--- however The atomic ratio of Ti (titanium) element in a metallic element and b a The atomic ratio of aluminum (aluminum) element in a metallic element, The atomic ratio of the carbon (C) element in a nonmetallic element and y x The atomic ratio of the nitrogen (N) element in a nonmetallic element, The atomic ratio [as opposed to / as opposed to / the atomic ratio of the oxygen (O) element in a nonmetallic element / in z] the sum of a metallic element in w / of a nonmetallic element is expressed. each -- a -- + -- b -- = -- one -- 0.8 -- > -- a -- > -- zero -- . -- four -- x -- + -- y -- + -- z -- = -- one -- 0.5 -- > -- x -- > -- zero -- one -- > -- y -- > -- zero -- . -- five -- 0.5 -- > -- z -- > -- zero -- 1.05 -- > -- w -- > -- 0.7 -- a relation -- it is ---] -- expressing -- having -- composite -- hard --

[Claim 9] the above-mentioned compound hard layer -- w(Cx (Tia, Alb, M1-a-b), Ny, Oz) [--- however The atomic ratio of Ti (titanium) element in a metallic element and b a The atomic ratio of aluminum (aluminum) element in a metallic element, M expresses at least one sort in 4a and 5a of a periodic table, 6a group element, and Si, Mn, Mg and B. The atomic ratio of the carbon (C) element in a nonmetallic element and y x The atomic ratio of the nitrogen (N) element in a nonmetallic element, The atomic ratio [as opposed to / as opposed to / the atomic ratio of the oxygen (O) element in a nonmetallic element / in z] the sum of a metallic element in w / of a nonmetallic element is expressed. each -- 0.8 -- > -- a -- > -- zero -- . -- four -- 0.6 -- > -- b -- > -- zero -- . -- two -- x -- + -- y -- + -- z -- = -- one -- 0.5 -- > -- x -- > -- zero -- one -- > -- y -- > -- zero -- . -- five -- 0.5 -- > -- z -- > -- zero -- 1.05 -- > -- w -- > -- 0.7 -- a relation -- it is ---] -- expressing -- having --

[Claim 10] The above-mentioned compound hard layer is a compound hard layer covering member given in any 1 term of the claims 1-9 which the metals of nickel, Co, W, Mo, aluminum, and Ti, these mutual alloys, and at least one sort of compound hard layer strengthening matter chosen from these intermetallic compounds contain in this compound hard layer.

[Claim 11] The above-mentioned compound hard layer is a compound hard layer covering member given in any 1 term of the claims 1-10 by which the columnar crystal which comes to be pillar-shaped is perpendicularly contained to the front face of the above-mentioned base material.

[Claim 12] The above-mentioned compound hard layer is a compound hard layer covering member given in any 1 term of the claims 1-11 which the content of aluminum element to the sum content of the metallic element of Ti and aluminum in this compound hard layer is increasing from the front face of the above-mentioned base material toward the front face of this compound hard layer.

[Claim 13] The above-mentioned compound hard layer is a compound hard layer covering member given in any 1 term of the claims 1-12 which the surface roughness of this compound hard layer becomes from 0.1 micrometers or less by the mean surface roughness by Ra of JIS.

[Claim 14] A compound hard layer covering member given in any 1 term of the claims 1-13 to which the thin layer which becomes by the intermetallic compound containing the metal of Ti and/or aluminum, the alloy of Ti and aluminum, Ti, and/or aluminum intervenes by the layer thickness of 1 micrometer or less between the above-mentioned compound hard layer and the above-mentioned base material.

[Claim 15] A compound hard layer covering member given in any 1 term of the above-mentioned claims 1-14 is a compound hard layer covering member used as a cutting tool.

[Claim 16] The above-mentioned cutting tool is a compound hard layer covering member according to claim 15 to which the layer thickness of the above-mentioned compound hard layer is decreasing toward the ridgeline section.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[The technical field to which invention belongs] this invention relates to the compound hard layer covering member with which the enveloping layer containing the compound hard layer which becomes by the ***** aluminum inclusion conjugated compound on the base material of a metal, an alloy, a sintered alloy, a ceramic sintered compact, or a super-elevated-temperature hyperbaric-pressure sintered compact was covered.

[0002]

[Description of the Prior Art] A hard layer is covered from the former using a chemical vapor deposition (henceforth "CVD"), a physical vapor deposition (henceforth "PVD"), or a plasma CVD method on the base material of a metal, an alloy, a sintered alloy, a ceramic sintered compact, or a super-elevated-temperature hyperbaric-pressure sintered compact, and the hard layer covering member which used the base material and the hard layer effectively has been used. Now, the quality of the material of the hard layer in the hard layer covering member currently used can mention Ti element inclusion hard layers, such as a nitride of Ti, a charcoal nitride, and carbide, Ti-aluminum element inclusion compound hard layers, such as a compound nitride of inclusion of Ti and aluminum, and a compound charcoal nitride, and an aluminum-oxide hard layer as an example of representation.

[0003] Among these hard layer covering members, a Ti-aluminum element inclusion compound hard layer is covered on a base material, the property of a Ti-aluminum element inclusion compound hard layer is pulled out effectively, and many compound hard layer covering members which were going to attain the longevity life are proposed. To among these, the typical thing proposed about making a longevity life attain from the crystal structure of a compound hard layer A publication-number 209335 [eight to] official report, a publication-number 291353 official report, a publication-number 295204 [nine to] official report, A publication-number 300105 [nine to] official report, a publication-number 300106 [nine to] official report, a publication-number 323204 [nine to] official report, A publication-number 323205 [nine to] official report, a publication-number 76407 [ten to] official report, a publication-number 76408 [ten to] official report, There are a publication-number 1762 [11 to] official report, a publication-number 131214 [11 to] official report, a publication-number 131215 [11 to] official report, a publication-number 131216 [11 to] official report, and a publication-number 131217 [11 to] official report. Moreover, a Provisional-Publication-No. 28478 [52 to] official report is in the typical thing proposed about the covered cemented carbide which covered not a Ti-aluminum element inclusion compound hard layer but Ti element inclusion compound hard layer.

[0004]

[Problem(s) to be Solved by the Invention] The inside of the advanced-technology reference about the compound hard layer covering member which covered the Ti-aluminum element inclusion compound hard layer, A publication-number 209335 [eight to] official report, a publication-number 295204 [nine to] official report, a publication-number 300105 [nine to] official report, A publication-number 300106 [nine to] official report, a publication-number 323204 [nine to] official report, a publication-number 323205 [nine to] official report, In a publication-number 76407 [ten to] official report, a publication-number 76408 [ten to] official report, a publication-number 131215 [11 to] official report, and a publication-number 131217 [11 to] official report The ratio of the peak height of the crystal face to the peak height (200) of the crystal face in the X-ray diffraction (111) of a Ti-aluminum element inclusion compound hard layer is indicated about the covering member containing the requirements for a configuration which are 1 or more, 1.5 or more, and 2 or more. Moreover, it is indicated about the covering member which includes the requirements for a configuration whose ratios of the peak height of the crystal face to the peak height (200) of the crystal face in the X-ray diffraction (111) of a Ti-aluminum element inclusion compound hard layer are two or less among advanced-technology reference in a publication-number 291353 [nine to] official report, a publication-number 131214 [11 to] official report, and a publication-number 131216 [11 to] official report.

[0005] moreover, in a publication-number 317123 [ten to] official report The inside of the diffraction peak of the compound hard layer represented by N in the X-ray diffraction which makes Cu and K alpha rays a line source (Ti, aluminum). About the compound hard layer member containing the requirements for a configuration from which the crystal face serves as the highest diffraction peak height, further (200) In a publication-number 1762 [11 to] official report It is indicated about the compound hard layer covering member which makes the highest diffraction peak height the requirements for a configuration at the angle of diffraction in 42.5 - 44.5 degrees (2theta) among the diffraction peaks of this compound hard layer in the X-ray diffraction which makes Cu and K alpha rays a line source.

[0006] Having made the stable cutting and the longevity life possible is indicated by the open patent official report of these 15 affairs by exhibiting suppression of the intergranular fracture in a compound hard layer, the enhancement in the adhesion of a base material and a compound hard layer, and wear-resistant enhancement in consideration of the residual compression stress in the compound hard layer in a Ti-aluminum element inclusion compound hard layer, or the crystal orientation in a compound hard layer. However, the compound hard layer covering member indicated by this official report of these 15 affairs From the defect of a crystal and asymmetry which exist in a compound hard layer not being considered It cannot be satisfied with the intensity of the compound hard layer [itself], and abrasion resistance, and cannot be satisfied with the adhesion with other layers which, as a result, adjoin the adhesion of a base material and a compound hard layer, and a compound hard layer. The variation in a life is large, and when it uses as a cutting tool, it being stabilized to a wide range field from a low-temperature field to an elevated-temperature field, and obtaining a longevity life has the problem are difficult.

[0007] In addition, although it is not the advanced-technology reference about a Ti-aluminum element inclusion

compound hard layer, the X-ray diffraction line in the crystal face (200) of a hard layer is indicated by the Provisional-Publication-No. 28478 [52 to] official report by 2theta about the hard layer member whose half peak width is 0.4 degrees or more. The hard layer member of an indication in this official report is a hard layer by PVD, and the half peak width is expressing the difference with the hard layer by CVD. From not considering the defect of the crystal of a hard layer, and asymmetry, like the compound hard layer mentioned above, the intensity of a hard layer, it cannot be satisfied with abrasion resistance and adhesion, and the variation in a life is large, and when it uses as a cutting tool, it being stabilized to a wide range field from a low-temperature field to an elevated-temperature field, and obtaining a longevity life has the problem are difficult.

[0008] this invention is what solved the above troubles. specifically The defect, asymmetry, the crystal structure, and crystal orientation of the crystal of the compound hard layer which becomes by the conjugated compound containing ~~*****~~ aluminum are considered. Especially, expand the use field as a cutting tool and the variation in the property of a compound hard layer is suppressed. The compound hard layer with high toughness, high degree-of-hardness nature, abrasion resistance, oxidation resistance, a thermal shock resistance, deficit-proof nature, and welding-proof nature. It aims at offer of a compound hard layer covering member which made the longevity life attain much more by considering as the compound hard layer which raised especially high intensity and a peeling resistance, and raised the welding-proof nature with ~~**~~-ed material.

[0009]

[Means for Solving the Problem] The research about membrane formation of the hard layer concerning [this invention persons] CVD, PVD, and plasma PVD. The result which continued at the long period of time and has performed the research about the hard layer especially by PVD, If perform enhancement in the plasma density at the time of membrane formation of the compound hard layer of Ti-aluminum inclusion, and enhancement in an ionization efficiency, it is made to gaseous-phase-method-grow epitaxially further and orientation of the crystal is carried out the optimum also in a hard layer The defect of the crystal of a compound hard layer is suppressed [that the asymmetry in a compound hard layer is eased uniformly,]. Since the compound hard layer of a fine crystal is obtained, it is enabled to raise the intensity, the abrasion resistance, the oxidation resistance, and thermal resistance of the compound hard layer [itself]. With the 1st knowledge that the enhancement in the adhesion with a compound hard layer, a base material or a substratum layer, or an outer layer becomes remarkable, in this way, in resembling the crystal with a perfect hard layer It comes to acquire the 2nd knowledge that it is simple to judge from the height ratio and half-peak-width ratio of an at least two diffraction lines peak including the highest peak in X-ray diffraction. Based on these knowledge, it comes to complete this invention.

[0010] The compound nitride in which the compound hard layer covering member of this invention contains titanium and aluminum on the surface of a base material. It is covered as an enveloping layer of the monolayer containing at least one sort of compound hard layers in compound carbide, a compound charcoal nitride, a compound nitric oxide, a compound carbonation object, and a compound charcoal nitric oxide, or a laminating. When X-ray diffraction is carried out from the front face of this compound hard layer using a copper target (200) When the peak height of the crystal face is set to $h(200)$ and the peak height of the crystal face (111) is set to $h(111)$ It consists of $h(200) / h(111) \geq 4.0$, and when the half peak width of the peak of the ~~**~~ (200) crystal face is set to $d(200)$ and the half peak width of the peak of the ~~**~~ (111) crystal face is set to $d(111)$, it consists of $1.5 \geq d(200) / d(111) \geq 0.8$.

[0011] By the compound hard layer covering member of this invention strengthening orientation of the crystal face in the compound hard layer (200) containing titanium and aluminum using gaseous-phase method epitaxial growth, and suppressing the asymmetry in a compound hard layer as much as possible While the intensity of the compound hard layer [itself] and toughness were raised, when the ~~*****~~ effect that abrasion resistance is also excellent is exhibited and X-ray diffraction is carried out from the front face of a compound hard layer using a copper target When are set to $h(200) / h(111) < 4.0$ and it is set to $d(200) / d(111) > 1.5$, or $d(200) / d(111) < 0.8$ (200) Since the stacking tendency to the crystal face is weak, the defect in a layer and asymmetry become large and the above-mentioned ~~*****~~ effect becomes weak, it is determined as the above peak-height ratios and a half-peak-width ratio.

[0012]

[Embodiments of the Invention] The base material in the compound hard layer covering member of this invention It is possible to use it, if it is the material or matter which can bear the temperature heated when covering an enveloping layer. specifically For example, the metal member represented by stainless steel, a heat-resistant alloy, high-speed steel, a die steel, Ti alloy, and aluminum alloy. A cemented carbide, a cermet, the sintered alloy represented by the P / M high speed steel, an aluminum₂O₃ system sintered compact, an Si₃N₄ system sintered compact, a sialon system sintered compact, ZrO₂ system sintered compact, the ceramic sintered compact represented by the silicon-carbide system sintered compact, The super-elevated-temperature hyperbaric-pressure sintered compact represented by a cubic-boron-nitride system sintered compact and the diamond system sintered compact can be mentioned. Among these, when making into a base material the material or matter used as the tool for cutting, or a tool for antifriction from the former, it is desirable from the effect of the enhancement in a life as the tool for covering cutting or a tool for covering antifriction becoming high.

[0013] In making a cemented carbide into a base material among these base materials The joint phase which a principal component becomes with Co and/or nickel 3 - 15 % of the weight, If it is made the cemented carbide containing at least one sort of hard phases chosen out of 4a and 5a of the tungsten carbide of the remainder or a tungsten carbide, and a periodic table, the carbide of 6a group metal, charcoal nitrides, and these mutual solid solutions It is especially desirable from enabling it to exhibit properties, such as an intensity of a base material, toughness, and abrasion resistance, and the property of a compound hard layer the optimum, and the enhancement effect in a life as a cutting tool becoming remarkable. The joint phase and hard phase which constitute the cemented carbide at this time can use what consists of a composition component of the joint phase contained in the conventional cemented carbide, and a hard phase.

[0014] Moreover, in making a ceramic sintered compact into a base material, when it makes into a base material the silicon-nitride system sintered compact which becomes 80 - 98 % of the weight from the sintering acid of the remainder about a silicon nitride and/or sialon, it is enabled to exhibit the adjustment of a base material and a compound hard layer the optimum from mechanical properties, such as an intensity of a base material, toughness, abrasion resistance, and thermal conductivity, and a physical characteristic, the mechanical property of a compound hard layer, and a physical characteristic, the enhancement effect in a life as a cutting tool becomes remarkable When the silicon nitride which constitutes the silicon-nitride system sintered compact at this time becomes by alpha silicon

nitride and/or beta silicon nitride. The case where sialon becomes by alpha sialon and/or beta sialon is sufficient. A sintering acid is what can use the matter contained in the silicon-nitride system sintered compact or the sialon system sintered compact from the former. as a concrete sintering acid For example, at least one sort of sintering acids chosen out of the oxide, the magnesium oxide, the oxidation hafnium, the zirconium oxide, the aluminum oxide, the aluminium nitride, the oxidation silicon, and these mutual solid solutions of rare earth elements, such as Y, La, Ce, and Dy, can be mentioned.

[0015] Furthermore, when making into a base material the super-elevated-temperature hyperbaric-pressure sintered compact represented by a diamond system sintered compact and the cubic-boron-nitride system sintered compact, it is that the case where it becomes with the diamond system sintered compact which consists of a diamond more than 70 volume % and a grain-boundary phase of the remainder, and the cubic-boron-nitride system sintered compact which consists of the cubic boron nitride and grain-boundary joint phase more than 20 volume % is desirable. Among these, the metal with which the diamond (it is hereafter described as "DIA") contains the diamond system sintered compact in the diamond system sintered compact of the former [phase / grain-boundary / of 85 - 98 volume % and the remainder]. It is specifically desirable from being able to exhibit both the properties of a base material and a compound hard layer the optimum, and the enhancement in a life as a cutting tool becoming remarkable, when at least one sort in Co, nickel, Fe, Si, and these mutual solid solutions is included, an alloy and. The grain-boundary joint phase of 35 - 95 volume % and the remainder a cubic-boron-nitride system sintered compact 4a and 5a of a periodic table, the carbide of 6a group element, [a cubic boron nitride] A nitride, a boride, the nitride of Si, Mg, and aluminum, a boride, oxides, and these mutual solid solutions, When consisting of at least one sort chosen from the metal of Co, nickel, Ti, and aluminum, the alloy, and the intermetallic compound, it is desirable from being able to exhibit both the properties of a base material and a compound hard layer the optimum, and the enhancement in a life as a cutting tool becoming remarkable.

[0016] When there is surface precision of a base material and surface precision of a base material was made high as a problem common to these base materials, and the surface precision of a compound hard layer also becomes high, for example, it is used as a cutting tool, it is desirable from frictional resistance becoming low, the dry area of a compound hard layer front face and a *-ed material front face being suppressed, and the enhancement effect in a life becoming high. The surface precision of a base material has desirable 0.1 micrometers or less at Ra which is a center line average of roughness height in the surface roughness specified to JIS B0601, and, as for more desirable one, Ra consists of 0.05 micrometers or less.

[0017] The configuration of the enveloping layer containing the compound hard layer covered by these base-material front faces The substratum layer which adjoins a base material and is covered for the purpose of adhesion, the interlayer who becomes by the compound hard layer in this invention which adjoins this substratum layer and is covered, On the configuration which carries out a laminating more than two-layer, and a concrete target, the outermost layer covered the distinction before and behind use on the outer layer which adjoins this interlayer and is covered, and the front face of this outer layer, and for the purpose of an ornament A configuration for example, the enveloping layer covered one by one on the surface of a base material -- a base material -- the laminating which consists of the - substratum layer-compound hard layer (interlayer)-outer-layer-outermost layer The configuration of a laminating which consists of a base-material-substratum layer-compound hard layer (interlayer)-outer layer, the configuration which consists of a laminating of a base-material-substratum layer-compound hard layer (interlayer), The configuration of a laminating which consists of the base-material-substratum layer-compound hard layer (interlayer)-outermost layer, The configuration of a laminating which consists of the base-material-compound hard layer (interlayer)-outer-layer-outermost layer, the configuration of a laminating which consists of a base-material-compound hard layer (interlayer)-outer layer, the configuration which consists of a laminating of the base-material-compound hard layer (interlayer)-outermost layer, or the configuration of a base-material-compound hard layer (interlayer) can be mentioned. Among these, it is desirable from that there is no complicatedness of the process at the time of a manufacture in a configuration of covering a compound hard layer directly on a base-material front face, becoming compaction of process time, and the variation on quality control decreasing.

[0018] A substratum layer becomes with a metal, an alloy, an intermetallic compound, or metallic compounds among these enveloping layers. specifically For example, metals [of Ti aluminum, nickel, Co, and W], these mutual alloys, Ti-aluminum, Ti-nickel, Ti-Co, aluminum-nickel, aluminum-Co, and Co-W, Ti-aluminum-nickel, the intermetallic compound of Ti-aluminum-Co, 4a and 5a of a periodic table, the carbide of 6a group metal, a nitride, a carbonation object, The case where it becomes by at least one sort of monolayers or the multilayer chosen out of a nitric oxide and the metallic compounds of these mutual solid solutions can be mentioned. An outer layer specifically Moreover, for example, 4a and 5a of a periodic table, carbide of 6a group metal, A nitride, a carbonation object, nitric oxides, these mutual solid solutions, the oxide of aluminum, a nitride, an acid nitride, a diamond, hard carbon (called diamond-like carbon), The case where it becomes by at least one sort of monolayers or the multilayer chosen from a cubic boron nitride, hard boron nitrides, and two or more sorts of such mixture can be mentioned. Furthermore, specifically, the outermost layer can mention the case where it becomes by at least one sort of monolayers or the multilayer chosen from the nitride of 4a, 5a, and 6a group metal, a charcoal nitride, nitric oxides, and these mutual solid solutions that what is necessary is just the enveloping layer in which the distinction before and behind use has easy color, and an enveloping layer with a decorative effect.

[0019] The compound hard layer used as the main point of this invention has the case of a configuration of being covered as an interlayer like the configuration of the above-mentioned enveloping layer, when it is the configuration with which an above-mentioned substratum layer and an above-mentioned compound hard layer are covered by the base-material front face when becoming with the configuration with which only the compound hard layer was covered by the base-material front face, if another representation is carried out when the enveloping layer itself becomes by the compound hard layer. If a chemical formula indicates the composition component of this compound hard layer, and membranous quality as concrete instantiation N, C (C (Ti, aluminum), N) (Ti, aluminum), (Ti, aluminum) (Ti, aluminum) (C (Ti, aluminum), N, O) (N, O) (C (Ti, aluminum), O) N, C (C (Ti, aluminum), M) N (Ti, aluminum, M), (Ti, aluminum, M) (Ti, aluminum, M) And (Ti, aluminum, M) (C, N, O) can mention the case where it becomes by at least one sort of monolayers or the laminating chosen out of inside (C (Ti, aluminum), M), O), (N, O) (However, the case of M where express one or more sorts of the element of the metal except Ti and aluminum and a semimetal, and it consists of at least one sort in 4a and 5a of a periodic table, 6a group element, rare earth elements, Mn element, Mg element, Si element, and B element especially is desirable)

[0020] When the metallic element contains only Ti and aluminum, these compound hard layers compound hard layer [of w which can be expressed with the following chemical formula (Cx, Ny, Oz) (Tia, Alb)] [-- however The atomic ratio of

Ti (titanium) element in a metallic element and b a The atomic ratio of aluminum (aluminum) element in a metallic element, The atomic ratio of the carbon (C) element in a nonmetallic element and y x The atomic ratio of the nitrogen (N) element in a nonmetallic element, The atomic ratio [as opposed to / as opposed to / the atomic ratio of the oxygen (O) element in a nonmetallic element / in z] the sum of a metallic element in w / of a nonmetallic element is expressed, each -- a -- + -- b -- = -- one -- 0.8 -- >= -- a -- >= -- zero . -- four -- x -- + -- y -- + -- z -- = -- one -- 0.5 -- >= -- x -- >= -- zero -- one -- >= -- y -- >= -- zero . -- five -- 0.5 -- >= -- z -- >= -- zero -- 1.05 -- >= -- w -- >= -- 0.7 -- a relation -- it is --] -- becoming -- a case -- **** -- Moreover it is distorted and is excelling in the intensity of the compound hard layer / itself /, abrasion resistance, and toughness, and] desirable from a defect being excellent in a peeling resistance few.

[0021] moreover, in becoming by the compound hard layer containing metallic elements other than Ti and aluminum compound hard layer [of w which can be expressed with the following chemical formula (C_x, N_y, O_z) (Ti_a, Al_b, M_{1-a-b})] [-- however The atomic ratio of Ti (titanium) element in a metallic element and b a The atomic ratio of aluminum (aluminum) element in a metallic element, M 4a and 5a of a periodic table, 6a group element, rare earth elements, Si element, Mn element, At least one sort in Mg element and B element is expressed. x The atomic ratio of the carbon (C) element in a nonmetallic element, The atomic ratio of the nitrogen (N) element in a nonmetallic element and z y The atomic ratio of the oxygen (O) element in a nonmetallic element, w -- a metallic element -- the sum -- receiving -- a nonmetallic element -- an atomic ratio -- expressing -- each -- 0.8 -- >= -- a -- >= -- zero -- . -- four -- 0.6 -- > -- b -- > -- zero -- . -- two -- x -- + -- y -- + -- z -- = -- one -- 0.5 -- >= -- x -- >= -- zero -- one -- >= -- y -- >= -- zero -- . -- five -- 0.5 -- >= -- z -- >= -- zero -- 1.05 -- >= -- Moreover it is distorted and is [excelling in abrasion resistance and toughness, and] desirable from a defect being excellent in a peeling resistance few.

[0022] When the compound hard layer strengthening matter which becomes the interface of the crystal grain child of a compound hard layer from a metallic element contains these compound hard layers in the ultralow volume, they are that the intensity of the compound hard layer / itself / and toughness are much more excellent, that asymmetry is eased, and] desirable from the enhancement in a peeling resistance becoming remarkable. The compound hard layer strengthening matter at this time is [raising the adjustment of a base material and a compound hard layer, and] desirable from raising adhesion, when consisting of a metallic element which constitutes the base material. Although it is also possible to form by plating, the vacuum deposition method, etc. and to diffuse this, before covering an enveloping layer, when this compound hard layer strengthening matter diffuses the metallic element which constitutes the base material in a compound hard layer, it is a desirable thing from being obtained simply.

[0023] The content of aluminum element to the sum content of the metallic element of Ti and aluminum in a compound hard layer is increasing these compound hard layers from the base-material front face toward the front face of a compound hard layer. If another representation is carried out, the content of Ti element will increase from the front face of a compound hard layer toward a base-material front face. When it is made the compound hard layer of the so-called inclination composition, the adhesion of a base material and a compound hard layer is excellent. It is desirable from excelling in the intensity of the compound hard layer [itself], and toughness, and the oxidation resistance of that a defect, asymmetry, and residual stress decrease and a compound hard layer front face, abrasion resistance, and a corrosion resistance being excellent. Though the increase in aluminum element at this time and Ti element has increase and decrease in micro stair-like and in the shape of [of a saw] a blade, when it increases to a macro target gradually, the case where it is increasing continuously the shape of a parabola and in the shape of a straight line is sufficient as it.

[0024] This compound hard layer is a desirable thing from the collapse-proof intensity from the front face of a compound hard layer improving, and excelling in a peeling resistance and minute-proof chipping nature, when the columnar crystal which grew perpendicularly pillar-shaped to the base-material front face is contained, judging from the structure of the compound hard layer [itself]. Specifically, the compound hard layer containing this columnar crystal can illustrate the case where minute amount inclusion of the compound hard layer strengthening matter mentioned above in each of these granular crystals and a columnar crystal is carried out, when the whole compound hard layer becomes in the layer of a columnar crystal, it becomes in the intermingled layer of a granular crystal and a columnar crystal and it becomes by the laminating of the layer of a granular crystal, and the layer of a columnar crystal. Among these, when the compound hard layer strengthening matter contains below 1 volume % preferably below 3 volume % to the sum of a compound hard layer and the compound hard layer strengthening matter, while it excels in the collapse-proof intensity of **** both [a perpendicular direction and / horizontal] a front face, and compressive strength-proof, it is desirable from the ability to demonstrate the ***** effect of excelling also in abrasion resistance. [of a compound hard layer] [both]

[0025] As for these compound hard layers, it is desirable to consider the structure of the compound hard layer [itself] by in which position of the configuration of an enveloping layer mentioned above it exists. as a configuration of this enveloping layer When covering a direct compound hard layer to a base material, after covering a substratum layer to a base material, The compound hard layer member by the 1st configuration for which a compound hard layer is covered in a substratum layer, and the front face of a compound hard layer is used in the status contact other matter (for example, **ed material in a cutting tool). When an outer layer is covered on the front face of a compound hard layer, an outer layer and the outermost layer can be covered on the front face of a compound hard layer, and it can divide roughly into the compound hard layer member by the 2nd configuration for which the front face of a compound hard layer is used in the status that it does not contact other matter and directly.

[0026] among these, in the case of the compound hard layer member by the 1st configuration If the front face of a compound hard layer sets 0.1 micrometers or less to 0.05 micrometers or less preferably by center line average-of-roughness-height Ra in the surface roughness specified to JIS B0601 When it is used as a cutting tool, from that the trauma to **ed material is eased, that a cutting force is eased, and issue of a chip becoming easy, it becomes long lasting and is much more desirable thing. Moreover, also in the compound hard layer member by the 2nd configuration, although it changes with the layer thickness of an outer layer, or sum layer thickness of an outer layer and the outermost layer, when the surface roughness of a compound hard layer is made above, the surface roughness of an outer layer and the outermost layer is also a desirable thing from it being smooth, becoming flat and being able to demonstrate the same effect as ****.

[0027] Although the layer thickness of each class which constitutes an enveloping layer is chosen by the configuration of intended use, a configuration, and an enveloping layer, it In the case of the compound hard layer member by the 1st above-mentioned configuration In making 1-20 micrometers of substratum layers the layer thickness of a compound hard layer mainly intervene for the purpose of adhesion in this case by the subject of an enveloping layer serving as a

compound hard layer When layer thickness of a substratum layer is set to 0.2–2 micrometers, it is desirable from the intensity, the abrasion resistance, the toughness, and peeling resistance of the enveloping layer [itself]. Moreover, in becoming by the compound hard layer member by the 2nd above-mentioned configuration, when it sets [the layer thickness of a substratum layer / the layer thickness of 0.2–2 micrometers and a compound hard layer] layer thickness of 1–10 micrometers and the outermost layer to 0.5–2 micrometers for 1–10 micrometers and the layer thickness of an outer layer, it is desirable from the ability to exhibit the property of each membrane layer the optimum. When a start and substratum layer, an outer layer, and the outermost layer become by the stoichiometric composition about the compound hard layer explained in full detail above, the case where it becomes by the non-stoichiometric composition is sufficient, and it consists of a non-stoichiometric composition in many cases substantially.

[0028] The compound hard layer member of this invention which becomes with the above gestalt It is a thing usable for various kinds of intended use. specifically for example, as the intermittence cutting tool which a lathe-turning tool, a milling cutter tool, a drill, the cutting tool represented by the end mill, especially **ed material are a casting and steel, and needs shock resistance, or a rotation cutting tool As tools for antifriction, such as disconnection blades, such as a slitter, and a decision blade, from mold tools, such as a die and punch It is usable as an engineering-works construction tool represented by the disconnection tool used for a mine, a passage, civil engineering works, etc., a digging tool, a **** tool, and the spallation tool as tools for cauterization-proof antifriction, such as a nozzle and a tool with **. Among these, the compound hard layer member of this invention is a desirable thing from exhibiting the property of a compound hard layer the optimum, when using it as cutting tools, such as rotation cutting tools, such as a cutting tool with which temperature, a friction, a thermal shock, compression impact, etc. serve as a severe condition most in micro especially a drill, and an end mill, and a throwaway tip. When using this compound hard layer member as a cutting tool, when the layer thickness of a compound hard layer is formed so that it may decrease toward the ridgeline section formed in the cutting edge of a cutting tool, it is a desirable thing from excelling in a peeling resistance and minute chipping nature. Moreover, being formed so that the layer thickness of the enveloping layer containing these compound hard layers may decrease toward the ridgeline section formed in the cutting edge of a cutting tool also makes the same effect caused, and it is a desirable thing.

[0029] The metal member represented by the stainless steel by which the compound hard layer covering member of this this invention is marketed from the former, a heat-resistant alloy, high-speed steel, a die steel, Ti alloy, and aluminum alloy, A cemented carbide, a cermet, the sintered alloy represented by the P / M high speed steel, an aluminum2O3 system sintered compact, an Si3N4 system sintered compact, a sialon system sintered compact, ZrO2 system sintered compact, the ceramic sintered compact represented by the silicon-carbide system sintered compact, The super-elevated-temperature hyperbaric-pressure sintered compact represented by a cubic-boron-nitride system sintered compact and the diamond system sintered compact is made into a base material. Although an enveloping layer can be covered with the PVD, the CVD, or plasma CVD method currently performed from the former on a base material and it can produce after grinding the front face of this base material if needed and performing ultrasonic cleaning, organic solvent cleaning, etc. When it produces by the following technique, the property and adhesion of that the gaseous-phase epitaxial crystal growth and crystal orientation of that the enhancement in a plasma density and the enhancement in an ionization efficiency are attained and the compound hard layer [itself] become easy and a compound hard layer are a desirable thing from excelling more.

[0030] When the important characteristic feature is concretely explained in full detail as the manufacture technique for obtaining this compound hard layer covering member, the front face of a base material At least one sort of mechanical processes in blast processing currently performed from the former, peening processing, polishing processing, and barrel processing, At least one sort of chemical preparations in washing by the surface cauterization by the electrolytic etching by the acid or alkaline electrolytic solution, the acid solution, and the alkali solution or water, and the organic solution, When processing chosen from the art which performs this mechanical process and chemical preparation simultaneous or separately is performed, it is desirable from the ability to suppress [that the defect on the front face of a base material is removable, that the adhesion of a compound hard layer can be raised, that the asymmetry in a layer can be suppressed, and] the defect in a layer. Moreover, a base material is that it is desirable to add such the mechanical treatment and/or a chemical preparation, and heat treatment by low temperature, and to also raise an above-mentioned effect.

[0031] When covering a compound hard layer on the surface of a base material, it is especially desirable from adjustment of a compound hard layer being easy, when it is desirable to carry out by the PVD represented by a sputtering technique and the ion-plating method, among these it carries out by the magnetron sputtering technique or the arc-plasma ion-plating method. When a base material is arranged in the reaction container of an ion plating system, it specifically carries out bombardment processing of the base-material front face and bombardment processing by metallic element ion or bombardment processing by both metallic element ion and nonmetallic element ion is performed, it is desirable from an above-mentioned effect being raised. When you need a metal, an alloy, or the substratum layer of an intermetallic compound among the above-mentioned substratum layers and it especially gives ion bombardment containing metallic element ion, it is desirable from that formation of a substratum layer is easy, and the adhesion of a base material and a substratum layer becoming high.

[0032] The covering conditions of a compound hard layer need to attach importance to the influence of the equipment, such as the structure of a reaction container, and adjustment of a plasma, itself. specifically for example, the high voltage and (— using the equipment which carries out the plasma occurrence of the ion for the pulse-like high voltage and a RF with acceleration with the power of addition) by the case, and the equipment which can adjust the plasma by the magnetic field — In addition, the thing for which it is necessary to consider about arrangement of the ambient-pressure force in a reaction container, temperature, an arc discharge current . voltage, base-material bias voltage, and a sample etc. among these, and especially an arc discharge voltage is made high to the conventional conditions, Rotations, vertical movement, etc. of making base-material bias voltage high and a sample are important requirements.

[0033]

[Operation examination 1] An operation examination explains the operation gestalt of this invention explained in full detail above as a still concrete example of representation. First, the examination which covered the direct compound hard layer on these base-material front faces is explained using the base material of a cemented carbide and the base material of a cermet which were produced through each process of the conventional combination, mixture, molding, and sintering. The base material 1 of the cemented carbide of SNGN120408 configuration — the base material 5, and the base material 6 of a cermet by ISO specification produced by Table 1 shown of the combination composition

component are used for a base material. A grinding process is given for the vertical side and periphery side of these base materials by the diamond wheel of 270#. After having given the -25° degree $\times 0.10\text{mm}$ honing to the edge-of-a-blade section by 400# diamond wheel and performing wet blast processing, washing processing, and xeransis processing for a front face further, the compound hard layer was covered with the arc ion plating system.

[0034] Processing conditions covered the compound hard layer, after carrying out bombardment processing of each base-material front face in a reaction container. The ambient-atmosphere: vacuum in a reaction container, base-material temperature: 873K, arc current: 70A, base-material bias voltage: -600V , and Ar gas bombardment performed bombardment processing. Quantity-of-gas-flow: 200 – 350SCCM in a reaction container, an evaporation-source: Ti-aluminum alloy, arc voltage: 200–300V, arc current: 150–200A, base-material temperature: 773–873K, and base-material bias voltage: -100 – -200V performed covering of a compound hard layer, and this invention article 1–6 which covered the compound hard layer on each front face of the base materials 1–6 shown in Table 1, and was shown in Table 2 was obtained. this invention article 1 the ambient atmosphere in a reaction container Among these, Ar-N₂-O₂ gas composition, Ar-N₂-CO gas composition, in addition this invention article 3–6 carry [this invention article 2] out by Ar-N₂ gas composition. to the evaporation source of this invention article 4 It carried out by having changed from the alloy with many Ti elements to the alloy with many aluminum elements, and carried out to the evaporation source of this invention article 5 by changing to the alloy with many alloy-Ti elements with many alloy-aluminum elements with many Ti elements.

[0035] The comparison article 1–4 which covered the compound hard layer as a comparison on each front face of the base materials 3–6 shown in Table 1, and was shown in Table 2 was obtained. The base-material front face of use in this invention article 1–6 of a **** [a base-material surface roughness] was [the base-material front face of use in the comparison article 1–4] $R_a=0.1$ – 0.05micrometer to $R_a=0.01$ – 0.005micrometer . Except for wet blast processing, others processed almost similarly base-material processing in the comparison article 1–4 among the base-material surface treatment of this invention article mentioned above. Moreover, covering of the compound hard layer in this comparison article 1–4 was processed almost similarly except having been referred to as arc voltage: 10–50V, arc current: 200–250A, and base-material bias voltage: -30 – -80V among the processing conditions of the compound hard layer of this invention article mentioned above. However, the evaporation source at the time of compound hard layer processing of the comparison article 1–4 used what has a fixed Ti-aluminum element ratio.

[0036] in this way, about each compound hard layer of the obtained this invention article 1–6 and the comparison article 1–4 A testing machine and the scratch testing machine which scratches and is equivalent to a hardness tester are used in X-ray diffraction equipment, a scanning electron microscope, a metaloscope, EDS equipment, and the Vickers hardness. It asked for the content of Ti and aluminum of h (200) by the X-ray diffraction from a compound hard layer front face / d [h (111) and] (200)/d (111), and a compound hard layer front face, the hardness of a compound hard layer front face, and the scratch intensity, and each result was shown in Table 2. In addition, the compound hard layer thickness of this invention article 1–6 and the comparison article 1–4 consisted of about 5–7 micrometers, the granularity of the compound hard layer front face of this invention article 1–6 was $R_a=0.015$ – 0.010micrometer mostly, and the granularity of the compound hard layer front face of the comparison article 1–4 was $R_a=0.15$ – 0.10micrometer mostly. Moreover, the oxygen element in a nonmetallic element of the compound hard layer of this invention article 1 was ***** not more than 1at% (Ti, aluminum) (N, O), the oxygen element in a nonmetallic element of the compound hard layer of this invention article 2 was a charcoal nitride not more than 2at% (Ti, aluminum) (N, C), and other compound hard layers were nitrides displayed by N (Ti, aluminum).

[0037] Subsequently, the following cutting conditions performed the wet intermittence cutting examination using this invention article 1–5 and the comparison article 1–3. The four slots ON round bar, cutting-speed: $150\text{m}/\text{min}$, and min, delivery: $0.3\text{mm}/\text{min}$, and rev, slitting: 2.0mm , tool configuration: SNGN120408, and the water soluble cutting oil performed the cutting conditions to S45C of the carbon steel material for **ed material: machine structures. When the chipping of a cutting edge and an enveloping layer exfoliated, the result of a wet intermittence cutting examination made the tool life the time of the amount of mean flank wears or the amount of groove wears becoming 0.3mm , found each time at that time which can be cut, and wrote it together to Table 2 as a life ratio to the life of the comparison article 1.

[0038]

[Table 1]

試料番号	焼結合金の組成成分 (配合時)	重量%
基材 1	97WC-3Co	
基材 2	91WC-3TaC-1TiC-5Co	
基材 3	88WC-2TaC-2TiC-8Co	
基材 4	88.5WC-1TaC-0.5NbC-2TiC-10Co	
基材 5	80WC-1Cr3C2-1VC-18Co	
基材 6	28TiC-26TiN-20WC-10TaC-1Mo2C-1ZrC-8Ni-8Co	

[0039]

[Table 2]

試料 番号		複合硬質膜の 結晶面ピーク		複 合 硬 質 膜 の成分比、特性			切削試験 寿命比
		高さ比: h (200)/(111)	半幅幅比: d (200)/(111)	Ti:Al 比	表面硬さ (HV)	スクラッチ 強度(N)	
本発明品	1	5.0	0.85	52 : 48	2780	70	2.5
	2	4.6	0.9	55 : 45	2950	75	2.8
	3	7.5	1.1	53 : 47	2900	90	5.2
	4	5.3	1.4	56 : 44	2920	86	4.8
	5	5.8	1.2	58 : 42	3000	80	3.5
	6	6.6	1.3	50 : 50	2880	83	なし
比較品	1	3.7	1.2	53 : 47	2910	43	1.0
	2	1.4	2.3	50 : 50	2950	34	0.8
	3	2.4	2.5	55 : 45	2850	40	1.1
	4	0.1	0.6	40 : 60	3100	25	なし

[0040]

[Operation examination 2] The cemented carbide of the base material 3 shown in Table 1 of the operation examination 1 was made into the base material, and this invention article 7-13 and the comparison article 5 which covered the enveloping layer of the laminating which contains more than two-layer [in a substratum layer, a compound hard layer, an outer layer, and the outermost layer] on the front face of this base material, and the comparison article 6 were obtained using the arc ion plating system. Among these, Ti substratum layer of this invention article 7 carried out Ti vacuum evaporation to the base-material front face, carried out the vacuum evaporation of the intermetallic-compound (Ti-aluminum) substratum layer of this invention article 8 simultaneously with bombardment processing by the evaporation source (Ti-aluminum), and deposited simultaneously with bombardment processing by Ti evaporation source Ti substratum layer of this invention article 9. Base-material processing of this invention article 7-13 and membrane formation of a compound hard layer were processed almost like this invention article 3-6 in the operation examination 1, and processed base-material processing of the comparison article 5 and the comparison article 6 and membrane formation of a compound hard layer almost like the comparison article 1 in the operation examination 1. Other substratum layers, outer layers, and outermost layers were processed by the almost conventional process.

[0041] in this way, about the substratum layer, the compound hard layer, the outer layer, and the outermost layer of each of the obtained this invention article 7-13, the comparison article 5, and the comparison article 6 It investigated like the operation examination 1, the configuration of each enveloping layer, each membraneous quality, and layer thickness were shown in Table 3, and the crystal-face peak-height ratio by the X-ray diffraction in a compound hard layer front face, a half-peak-width ratio, the element ratio of Ti and aluminum, surface hardness, and the scratch intensity were shown in Table 4. Moreover, about this invention article 7-13, the comparison article 5, and the comparison article 6, the cutting examination was performed like the cutting conditions of the operation examination 1, and it asked for each life ratio to the comparison article 5, and wrote together to Table 4. In addition, after surface investigation of a compound hard layer covers a compound hard layer, it is taken out from a reaction container and performed, and this invention article 10 heat-treated, diffused the joint phase in a base material in the enveloping layer, and made the substratum layer and the compound hard layer contain the joint phase of about 1 volume % after [all] a covering end. Moreover, the charcoal titanium-nitride layer of a substratum layer consists of a columnar crystal, and this invention article 11 is considered as a columnar-crystal crystal in consideration of a plasma density, radical reaction, and a membrane formation speed at the time of membrane formation of a compound hard layer.

[0042]

[Table 3]

試料 番号	被覆層の膜質および膜厚さ (μm)			
	下地層	複合硬質膜	外層	最外層
本発明品	7	0.5Ti	6(Ti,Al)N	なし
	8	0.5(Ti-Al)	6(Ti,Al)N	なし
	9	0.5Ti-2TiN	6(Ti,Al)N	なし
	10	1TiN	3(Ti,Al)N	5Al ₂ O ₃
	11	1TiN-3TiCN	3(Ti,Al)N	5Al ₂ O ₃
	12	1.5TiN	3(Ti,Al)N	2TiNO-3Al ₂ O ₃
	13	1.5TiN	4(Ti,Al)N	2TiCO-3Al ₂ O ₃
比較品5	1TiN	3(Ti,Al)N	5Al ₂ O ₃	なし
比較品6	1.5TiN	4(Ti,Al)N	2TiNO-3Al ₂ O ₃	1TiN

[0043]

[Table 4]

試料 番号		複合硬質膜の 結晶面ピーク		複 合 硬 質 膜 の成分比、特性			切削試験 寿命比
		高さ比 : h (200)/(111)	半価幅比 : d (200)/(111)	Ti : Al 比	表面硬さ (HV)	スクラッチ 強度(N)	
本 発 明 品	7	7.3	1.2	58 : 42	2900	55	5.7
	8	7.4	1.3	53 : 47	2950	62	4.8
	9	7.6	1.2	55 : 45	2900	78	5.5
	10	7.8	1.1	54 : 46	2910	95	7.1
	11	8.5	1.0	50 : 50	3000	88	6.5
	12	7.4	0.9	51 : 49	2890	73	5.1
	13	7.2	0.95	57 : 43	2920	70	4.7
比較品5		2.3	3.0	54 : 46	2900	35	1.0
比較品6		3.0	2.4	53 : 47	2930	42	1.3

[0044]

[Operation examination 3] The base material 7 of the ceramic sintered compact produced by 70 volume %aluminum2O3-30 volume %TiCN (combination composition), The base material 8 of the ceramic sintered compact produced by the 70 volume %aluminum2O3-30 volume %SiC whisker (combination composition), The base material 9 of the ceramic sintered compact produced by 96 volume %Si3N4-1 volume %MgO-1 volume %Y2O3-2 volume %HfO2 (combination composition), The base material 10 of the ceramic sintered compact produced by 90 volume %Si3N4-4 volume %AlN-4 volume %aluminum2O3-1 volume %MgO-1 volume %Y2O3 (combination composition) is used. After having processed the base material almost like this invention article 3-6 in the operation examination 1 and depositing Ti of about 1 micrometer layer thickness on each base-material front face, the compound hard layer was covered and this invention article 14-17 was obtained. Moreover, using a base material 7 and the base material 9 as a comparison, it processed almost like the comparison article 1 in the operation examination 1, and the comparison article 7 and the comparison article 8 were obtained.

[0045] In this way, about the compound hard layer of the obtained this invention article 14-17, the comparison article 7, and the comparison article 8, it investigated like the operation examination 1, and the crystal-face peak-height ratio by the X-ray diffraction in a compound hard layer front face, a half-peak-width ratio, the element ratio of Ti and aluminum, surface hardness, and the scratch intensity were shown in Table 5. moreover, this invention article 14, this invention article 15, and the comparison article 7 -- **ed material -- :FCD600 and cutting-speed:150m/min -- cutting deeply -- : -- 1.5mm, it sent and :0.2mm /, blade, tool configuration:SNGN120408, honing:0.15x-25 degree, and the dry-type milling cutter cutting examination were performed this invention article 16, this invention article 17, and the comparison article 8 -- **ed material -- :FCD250 and cutting-speed:600m/min -- cutting deeply -- : -- 3.0mm, it sent and :0.1mm /, blade, tool configuration:SNGN120412, honing:0.15x-25 degree, and the dry-type milling cutter cutting examination were performed Like the cutting examination in the operation examination 1, these dry-type milling cutter results made this invention article 14 and this invention article 15 the life ratio to the comparison article 7, and wrote together this invention article 16 and this invention article 17 to Table 5 as a life ratio to the comparison article 8. In addition, the compound hard layer thickness of this invention article 14-17, the comparison article 7, and the comparison article 8 was about 5 micrometers.

[0046]

[Table 5]

試料 番号		複合硬質膜の 結晶面ピーク		複 合 硬 質 膜 の成分比、特性			切削試験 寿命比
		高さ比 : h (200)/(111)	半幅幅比 : d (200)/(111)	Ti : Al 比	表面硬さ (HV)	スラフ 強度(N)	
本 発 明 品	1 4	0.8	0.96	80 : 40	2880	76	6.5
	1 5	6.9	1.05	82 : 98	2850	68	6.1
	1 6	12.5	1.1	48 : 52	3150	70	5.3
	1 7	13.4	1.2	45 : 55	3200	73	6.0
比較品 7	8.5	2.3	58 : 42	2950	31	1.0	
比較品 8	3.7	2.1	46 : 54	3130	32	1.0	

[0047]

[Operation examination 4] The base material 11 of the cubic-boron-nitride system sintered compact of the super-elevated-temperature hyperbaric pressure produced by 40 volume %cBN-5 volume %aluminum2O3-5 volume %AlN-10 volume %aluminum-10 volume %Mg-10 volume %B-20 volume %TiN (combination composition). The base material 12 of the cubic-boron-nitride system sintered compact of the super-elevated-temperature hyperbaric pressure produced by 85 volume %cBN-2 volume %Co-5 volume %aluminum-2 volume %Mg-6 volume %TiN (combination composition). The base material 13 of DIA system sintered compact of the super-elevated-temperature hyperbaric pressure produced by the 95 volume %DIA-2 volume %Co-2 volume %nickel-1 volume ZrC (combination composition). The base material 14 of DIA system sintered compact of the super-elevated-temperature hyperbaric pressure produced with 97 volume %DIA-1 volume %Co-1 volume %nickel-1 volume Mg (combination composition) is used. After having processed the base material almost like this invention article 3-6 in the operation examination 1, having carried out the vacuum evaporation of Ti of about 1 micrometer layer thickness to the front face of a base material 11 and the base material 12 and giving nickel electroless deposition of about 1 micrometer thickness to the front face of a base material 13 and the base material 14, the compound hard layer was covered and this invention article 18-21 was obtained. Moreover, using a base material 11 and the base material 13 as a comparison, it processed almost like the comparison article 1 in the operation examination 1, and the comparison article 9 and the comparison article 10 were obtained.

[0048] In this way, about the compound hard layer of the obtained this invention article 18-21, the comparison article 9, and the comparison article 10, it investigated like the operation examination 1, and the crystal-face peak-height ratio by the X-ray diffraction in a compound hard layer front face, a half-peak-width ratio, the element ratio of Ti and aluminum, surface hardness, and the scratch intensity were shown in Table 6. moreover, this invention article 18, this invention article 19, and the comparison article 9 -- **ed material -- :SCM415 (hardness:abbreviation HRC61) and cutting-speed:150m/min -- cutting deeply -- : -- 0.5mm, it sent, :0.1mm /, rev, tool configuration:T **** 160408, honing:0.15x-25 degree, and the periphery continuity dry-type lathe-turning examination were performed, and the result was written together to Table 6 Evaluation of this periphery continuity dry-type lathe-turning examination was performed like the cutting examination in the operation examination 1, and it expressed as a life ratio to the comparison article 9. In addition, the compound hard layer thickness of this invention article 18-21, the comparison article 9, and the comparison article 10 was about 5 micrometers.

[0049]

[Table 6]

試料 番号		複合硬質膜の 結晶面ピーク		複 合 硬 質 膜 の成分比、特性			切削試験 寿命比
		高さ比 : h (200)/(111)	半幅幅比 : d (200)/(111)	Ti : Al 比	表面硬さ (HV)	スラフ 強度(N)	
本 発 明 品	1 8	19.5	1.1	58 : 42	2960	85	8.2
	1 9	18.4	1.0	50 : 41	2930	83	8.8
	2 0	8.5	1.05	55 : 45	2960	76	なし
	2 1	8.0	0.95	54 : 46	2970	74	なし
比較品 9		3.1	2.0	55 : 45	2910	23	1.0
比較品 10		3.5	1.7	52 : 48	2940	25	なし

[0050]

[Effect of the Invention] The crystal growth according [the compound hard layer covering member of this invention] to a gaseous-phase method epitaxial and the compound hard layer by crystal orientation are covered, It is [that asymmetry by the compound hard layer itself and the defect are suppressed,] a fine crystal. It contrasts with the compound hard layer covering member which separated from a conventional compound hard layer covering member or a conventional this invention from it being the compound hard layer with which compound hard layer strengthening

matter, such as a metal of a columnar-crystal crystal and/or a minute amount, was contained by the case. Adhesion and a peeling resistance are very excellent to the matter contiguous to a base material, a compound hard layer, a substratum layer and a compound hard layer, a compound hard layer, and compound hard layers, such as an outer layer. The high toughness, the high intensity, the thermal resistance, the thermal shock resistance, the oxidation resistance, and abrasion resistance of the compound hard layer [itself] are excellent. As the result, for example, the thing for which the high toughness to which importance is attached, abrasion resistance, a thermal shock resistance, deficit-proof nature, oxidation resistance, and welding-proof nature improve notably, and reinforcement is attained as a cutting tool when it is used as a cutting tool. There is a remarkable effect that that efficient-ization in a cutting is attained and variation are small stable.

[Translation done.]

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(54)【発明の名称】 複合硬質膜被覆部材

(57)【要約】

【課題】従来のTi-Alを含む化合物でなる複合硬質膜の被覆部材は、複合硬質膜自体に多くの欠陥や歪みを有していることから、複合硬質膜と隣接する物質との密着性に劣ること、剥離しやすいこと、複合硬質膜自体の諸特性が劣ること、例えば切削工具として用いる場合には、複合硬質膜自体の欠陥や歪みから剥離、微小チップング、摩耗などが急速に発達しやすくなって、短寿命になるという課題がある。

【解決手段】基材の表面にチタンとアルミニウムとを含む複合窒化物、複合炭化物、複合炭窒化物、複合窒酸化物、複合炭酸化物、複合炭窒酸化物の中の少なくとも1種の複合硬質膜を含む単層または積層の被覆層として被覆されており、該複合硬質膜の表面から銅ターゲットを用いてX線回折したときに、(200)結晶面のピーク高さをh(200)とし、(111)結晶面のピーク高さをh(111)としたときに、 $h(200)/h(111) \geq 4.0$ からなり、該(200)結晶面のピークの半価幅をd(200)とし、該(111)結晶面のピークの半価幅をd(111)としたときに、 $1.5 \geq d$

$(200)/d(111) \geq 0.8$ からなる複合硬質膜被覆部材。

【特許請求の範囲】

【請求項1】基材の表面にチタンとアルミニウムとを含む複合窒化物、複合炭化物、複合炭窒化物、複合窒酸化物、複合炭酸化物、複合炭窒酸化物の中の少なくとも1種の複合硬質膜を含む単層または積層の被覆層として被覆されており、該複合硬質膜の表面から銅ターゲットを用いてX線回折したときに、(200)結晶面のピーク高さを $h(200)$ とし、(111)結晶面のピーク高さを $h(111)$ としたときに、 $h(200)/h(111) \geq 4.0$ からなり、該(200)結晶面のピークの半価幅を $d(200)$ とし、該(111)結晶面のピークの半価幅を $d(111)$ としたときに、 $1.5 \geq d(200)/d(111) \geq 0.8$ からなる複合硬質膜被覆部材。

【請求項2】上記基材は、主成分がCoおよび/またはNiでなる結合相を4~15重量%と、残部が炭化タングステンまたは炭化タングステンと周期律表の4a, 5a, 6a族金属の炭化物、炭窒化物およびこれらの相互固溶体から選ばれた少なくとも1種の硬質相を含有する超硬合金からなる請求項1に記載の複合硬質膜被覆部材。

【請求項3】上記基材は、窒化珪素および/またはサイアロンを80~98重量%と、残部が焼結助剤である窒化珪素系焼結体からなる請求項1に記載の複合硬質膜被覆部材。

【請求項4】上記基材は、立方晶窒化硼素を20~90重量%と、残部がTi, Al, Mg, Siの窒化物、硼化物およびこれらの相互固溶体の中から選ばれた少なくとも1種の粒界結合相とを含有する超高温高压焼結体からなる請求項1に記載の複合硬質膜被覆部材。

【請求項5】上記基材は、該基材の表面粗さがJIS規格のRaによる平均表面粗さで $0.1 \mu\text{m}$ 以下からなる請求項1~4のいずれか1項に記載の複合硬質膜被覆部材。

【請求項6】上記被覆層は、上記複合硬質膜でなる請求項1~5のいずれか1項に記載の複合硬質膜被覆部材。

【請求項7】上記複合硬質膜は、膜厚さが $1 \sim 15 \mu\text{m}$ でなる立方晶型結晶構造からなる請求項1~6のいずれか1項に記載の複合硬質膜被覆部材。

【請求項8】上記複合硬質膜は、 $(Ti_a, Al_b)(C_x, N_y, O_z)_w$ [ただし、aは金属元素中のTi(チタン)元素の原子比、bは金属元素中のAl(アルミニウム)元素の原子比、xは非金属元素中の炭素(C)元素の原子比、yは非金属元素中の窒素(N)元素の原子比、zは非金属元素中の酸素(O)元素の原子比、wは金属元素の合計に対する非金属元素の原子比を表し、それぞれが $a+b=1$ 、 $0.8 \geq a \geq 0.4$ 、 $x+y+z=1$ 、 $0.5 \geq x \geq 0$ 、 $1 \geq y \geq 0.5$ 、 $0.5 \geq z \geq 0$ 、 $1.05 \geq w \geq 0.7$ の関係にある]で表される複合硬質膜を含有する請求項1~7のいずれか1項に記載

の複合硬質膜被覆部材。

【請求項9】上記複合硬質膜は、 $(Ti_a, Al_b, M_{1-a-b})(C_x, N_y, O_z)_w$ [ただし、aは金属元素中のTi(チタン)元素の原子比、bは金属元素中のAl(アルミニウム)元素の原子比、Mは周期律表の4a, 5a, 6a族元素、Si, Mn, Mg, Bの中の少なくとも1種を表し、xは非金属元素中の炭素(C)元素の原子比、yは非金属元素中の窒素(N)元素の原子比、zは非金属元素中の酸素(O)元素の原子比、wは金属元素の合計に対する非金属元素の原子比を表し、それぞれが $0.8 \geq a \geq 0.4$ 、 $0.6 > b > 0.2$ 、 $x+y+z=1$ 、 $0.5 \geq x \geq 0$ 、 $1 \geq y \geq 0.5$ 、 $0.5 \geq z \geq 0$ 、 $1.05 \geq w \geq 0.7$ の関係にある]で表される複合硬質膜を含有する請求項1~7のいずれか1項に記載の複合硬質膜被覆部材。

【請求項10】上記複合硬質膜は、該複合硬質膜中にNi, Co, W, Mo, Al, Tiの金属、これらの相互合金、これらの金属間化合物の中から選ばれた少なくとも1種の複合硬質膜強化物質が含有されている請求項1~9のいずれか1項に記載の複合硬質膜被覆部材。

【請求項11】上記複合硬質膜は、上記基材の表面に対し、垂直方向に柱状でなる柱状結晶が含まれている請求項1~10のいずれか1項に記載の複合硬質膜被覆部材。

【請求項12】上記複合硬質膜は、該複合硬質膜中のTiとAlとの金属元素の合計含有量に対するAl元素の含有量が上記基材の表面から該複合硬質膜の表面に向かって増加している請求項1~11のいずれか1項に記載の複合硬質膜被覆部材。

【請求項13】上記複合硬質膜は、該複合硬質膜の表面粗さがJIS規格のRaによる平均表面粗さで $0.1 \mu\text{m}$ 以下からなる請求項1~12のいずれか1項に記載の複合硬質膜被覆部材。

【請求項14】上記複合硬質膜と上記基材との間に、Tiおよび/またはAlの金属、TiとAlの合金、Tiおよび/またはAlを含む金属間化合物でなる薄層が $1 \mu\text{m}$ 以下の膜厚さで介在されている請求項1~13のいずれか1項に記載の複合硬質膜被覆部材。

【請求項15】上記請求項1~14のいずれか1項に記載の複合硬質膜被覆部材は、切削工具として用いられる複合硬質膜被覆部材。

【請求項16】上記切削工具は、上記複合硬質膜の膜厚さが稜線部に向かって減少している請求項15に記載の複合硬質膜被覆部材。

【発明の詳細な説明】

【0001】

【発明の属する技術分野】本発明は、金属、合金、焼結合金、セラミックス焼結体または超高温高压焼結体の基材上にチタン-アルミニウム含有複合化合物でなる複合硬質膜を含む被覆層が被覆された複合硬質膜被覆部材に

関するものである。

【0002】

【従来の技術】従来から金属、合金、焼結合金、セラミックス焼結体または超高温高压焼結体の基材上に、化学蒸着法（以下、「CVD法」という）、物理蒸着法（以下、「PVD法」という）またはプラズマCVD法を利用して硬質膜を被覆し、基材と硬質膜とを有効に利用した硬質膜被覆部材が実用されてきている。現在、実用されている硬質膜被覆部材における硬質膜の材質は、Tiの窒化物、炭窒化物、炭化物などのTi元素含有硬質膜と、TiとAlを含有の複合窒化物、複合炭窒化物などのTi-Al元素含有複合硬質膜と、酸化アルミニウム硬質膜を代表例として挙げるができる。

【0003】これらの硬質膜被覆部材のうち、基材上に、Ti-Al元素含有複合硬質膜を被覆し、Ti-Al元素含有複合硬質膜の特性を有効に引き出して、長寿命を達成しようとした複合硬質膜被覆部材が多数提案されている。これらのうち、複合硬質膜の結晶構造から長寿命を達成させることについて提案されている代表的なものに、特開平8-209335号公報、特開平291353号公報、特開平9-295204号公報、特開平9-300105号公報、特開平9-300106号公報、特開平9-323204号公報、特開平9-323205号公報、特開平10-76407号公報、特開平10-76408号公報、特開平11-1762号公報、特開平11-131214号公報、特開平11-131215号公報、特開平11-131216号公報、および特開平11-131217号公報がある。また、Ti-Al元素含有複合硬質膜ではなく、Ti元素含有化合物硬質膜を被覆した被覆超硬合金について提案されている代表的なものに、特開昭52-28478号公報がある。

【0004】

【発明が解決しようとする課題】Ti-Al元素含有複合硬質膜を被覆した複合硬質膜被覆部材に関する先行技術文献のうち、特開平8-209335号公報、特開平9-295204号公報、特開平9-300105号公報、特開平9-300106号公報、特開平9-323204号公報、特開平9-323205号公報、特開平10-76407号公報、特開平10-76408号公報、特開平11-131215号公報、および特開平11-131217号公報には、Ti-Al元素含有複合硬質膜のX線回折における(111)結晶面のピーク高さに対する(200)結晶面のピーク高さの比が1以上、1.5以上または2以上である構成要件を含む被覆部材について開示されている。また、先行技術文献のうち、特開平9-291353号公報、特開平11-131214号公報および特開平11-131216号公報には、Ti-Al元素含有複合硬質膜のX線回折における(111)結晶面のピーク高さに対する(200)結

晶面のピーク高さの比が2以下である構成要件を含む被覆部材について開示されている。

【0005】また、特開平10-317123号公報には、Cu、K α 線を線源とするX線回折における(Ti, Al)Nに代表される複合硬質膜の回折ピークのうち、(200)結晶面が最高回折ピーク高さとなる構成要件を含む複合硬質膜部材について、さらに特開平11-1762号公報には、Cu、K α 線を線源とするX線回折における同複合硬質膜の回折ピークのうち、42.5~44.5度内の回折角(2 θ)に最高回折ピーク高さを構成要件とする複合硬質膜被覆部材について開示されている。

【0006】これら15件の公開特許公報には、Ti-Al元素含有複合硬質膜における複合硬質膜内の残留圧縮応力、または複合硬質膜内の結晶配向を考慮し、複合硬質膜内の粒界破壊の抑制、基材と複合硬質膜との密着性の向上、耐摩耗性の向上を発揮させることにより、安定した切削加工と長寿命を可能としたことが開示されている。しかしながら、これら15件の同公報に記載されている複合硬質膜被覆部材は、複合硬質膜に存在する結晶の欠陥および歪みに配慮されていないことから、複合硬質膜自体の強度、耐摩耗性に満足できなく、その結果基材と複合硬質膜との密着性および複合硬質膜と隣接する他の膜との密着性に満足できなく、寿命のパラツキが大きく、切削工具として実用したときに低温領域から高温領域まで広範囲の領域において、安定して長寿命を得ることが困難であるという問題を有している。

【0007】その他、Ti-Al元素含有複合硬質膜に関する先行技術文献ではないが、特開昭52-28478号公報には、硬質膜の(200)結晶面におけるX線回折線が2 θ で半価幅が0.4度以上である硬質膜部材について開示されている。同公報に開示の硬質膜部材は、PVD法による硬質膜であり、CVD法による硬質膜との相違を半価幅により表現しており、硬質膜の結晶の欠陥および歪みを配慮していないことから、上述した複合硬質膜と同様に硬質膜の強度、耐摩耗性および密着性に満足できなく、寿命のパラツキが大きく、切削工具として実用したときに低温領域から高温領域まで広範囲の領域において、安定して長寿命を得ることが困難であるという問題を有している。

【0008】本発明は、上述のような問題点を解決したもので、具体的には、チタン-アルミニウムを含む複合化合物でなる複合硬質膜の結晶の欠陥、歪み、結晶構造および結晶配向を配慮し、特に切削工具としての使用領域を拡大し、複合硬質膜の特性のパラツキを抑制し、高靱性、高硬度性、耐摩耗性、耐酸化性、耐熱衝撃性、耐欠損性、耐溶着性のある複合硬質膜、特に高強度および耐剥離性を高めて被削材との耐溶着性を向上させた複合硬質膜とすることにより一層長寿命を達成させた複合硬質膜被覆部材の提供を目的とするものである。

【0009】

【課題を解決するための手段】本発明者らは、CVD法、PVD法およびプラズマPVD法に関する硬質膜の成膜についての研究、特にPVD法による硬質膜についての研究を長期に亘って行ってきた結果、硬質膜の中でもTi-Al含有の複合硬質膜の成膜時におけるプラズマ密度の向上およびイオン化効率の向上を行い、さらに気相法エピタキシャル成長させて、結晶を最適に配向させると、複合硬質膜内の歪みが均一に緩和されること、複合硬質膜の結晶の欠陥が抑制されること、微細結晶の複合硬質膜が得られることから、複合硬質膜自体の強度、耐摩耗性、耐酸化性および耐熱性を向上させることが可能となり、複合硬質膜と基材または下地層や外層との密着性の向上が顕著になるという第1の知見と、このように硬質膜が完全な結晶に近似する場合には、X線回折における最高ピークを含めた少なくとも2本の回折線ピークの高さ比および半価幅比から判断することが簡易であるという第2の知見とを得るに至ったものである。これらの知見に基づいて、本発明を完成するに至ったものである。

【0010】本発明の複合硬質膜被覆部材は、基材の表面にチタンとアルミニウムとを含む複合窒化物、複合炭化物、複合炭窒化物、複合窒酸化物、複合炭酸化物、複合炭窒酸化物の中の少なくとも1種の複合硬質膜を含む単層または積層の被覆層として被覆されており、該複合硬質膜の表面から銅ターゲットを用いてX線回折したときに、(200)結晶面のピーク高さを $h(200)$ とし、(111)結晶面のピーク高さを $h(111)$ としたときに、 $h(200)/h(111) \geq 4.0$ からなり、該(200)結晶面のピークの半価幅を $d(200)$ とし、該(111)結晶面のピークの半価幅を $d(111)$ としたときに、 $1.5 \geq d(200)/d(111) \geq 0.8$ からなるものである。

【0011】本発明の複合硬質膜被覆部材は、気相法エピタキシャル成長を利用して、チタンとアルミニウムとを含む複合硬質膜における(200)結晶面の配向を強くし、複合硬質膜内の歪みを極力抑制することにより、複合硬質膜自体の強度、靱性を高めると共に、耐摩耗性もすぐれるというシナジー効果を発揮させたものであり、複合硬質膜の表面から銅ターゲットを用いてX線回折したときに、 $h(200)/h(111) < 4.0$ になる場合、 $d(200)/d(111) > 1.5$ または $d(200)/d(111) < 0.8$ になる場合には、(200)結晶面への配向性が弱く、膜内の欠陥および歪みが大きくなり、上述のシナジー効果が弱くなることから、上述のようなピーク高さ比および半価幅比と定めたものである。

【0012】

【発明の実施の形態】本発明の複合硬質膜被覆部材における基材は、被覆層を被覆するときに加熱する温度に耐

えることができる材料または物質ならば使用することが可能であり、具体的には、例えばステンレス鋼、耐熱合金、高速度鋼、ダイス鋼、Ti合金、Al合金に代表される金属部材、超硬合金、サーメット、粉末ハイスに代表される焼結合金、 Al_2O_3 系統結体、 Si_3N_4 系統結体、サイアロン系統結体、 ZrO_2 系統結体、炭化珪素系統結体に代表されるセラミックス焼結体、立方晶窒化硼系統結体、ダイヤモンド系統結体に代表される超高温高压焼結体を挙げることができる。これらのうち、従来から切削用工具または耐摩耗用工具として用いられている材料または物質を基材とする場合には、被覆切削用工具または被覆耐摩耗用工具としての寿命向上の効果が高くなることから、好ましいことである。

【0013】これらの基材のうち、超硬合金を基材とする場合には、主成分がCoおよび/またはNiでなる結合相を3~15重量%と、残部の炭化タングステンまたは炭化タングステンと周期律表の4a、5a、6a族金属の炭化物、炭窒化物およびこれらの相互固溶体から選ばれた少なくとも1種の硬質相とを含有する超硬合金にすると、基材の強度、靱性および耐摩耗性などの特性と複合硬質膜の特性とを最適に発揮させることが可能となり、切削用工具としての寿命向上効果が顕著となることから、特に好ましいことである。このときの超硬合金を構成している結合相および硬質相は、従来の超硬合金に含有されている結合相および硬質相の組成成分からなるものを採用できる。

【0014】また、セラミックス焼結体を基材にする場合には、窒化珪素および/またはサイアロンを80~98重量%と、残部の焼結助剤からなる窒化珪素系統結体を基材にすると、基材の強度、靱性、耐摩耗性、熱伝導性などの機械的特性および物理的特性と複合硬質膜の機械的特性および物理的特性から、基材と複合硬質膜との整合性を最適に発揮させることが可能となり、切削用工具としての寿命向上効果が顕著となり、特に好ましいことである。このときの窒化珪素系統結体を構成している窒化珪素が α 窒化珪素および/または β 窒化珪素でなる場合、サイアロンが α サイアロンおよび/または β サイアロンでなる場合でもよく、焼結助剤が従来から窒化珪素系統結体やサイアロン系統結体に含有されている物質を用いることができるものであり、具体的な焼結助剤としては、例えばY、La、Ce、Dy、などの希土類元素の酸化物、酸化マグネシウム、酸化ハフニウム、酸化ジルコニウム、酸化アルミニウム、窒化アルミニウム、酸化珪素およびこれらの相互固溶体から選ばれた少なくとも1種の焼結助剤を挙げることができる。

【0015】さらに、ダイヤモンド系統結体、立方晶窒化硼系統結体に代表される超高温高压焼結体を基材にする場合には、70体積%以上のダイヤモンドと残部の粒界相とからなるダイヤモンド系統結体、ならびに20体積%以上の立方晶窒化硼と粒界結合相とからなる立

方晶窒化硼系系統結体でなる場合が好ましいことである。これらのうち、ダイヤモンド系統結体は、ダイヤモンド（以下、「DIA」と記す）が85～98体積%と残部の粒界相が従来のダイヤモンド系統結体に含有されている金属、合金、具体的には、Co、Ni、Fe、Siおよびこれらの相互固溶体の中の少なくとも1種を含む場合には、基材と複合硬質膜との両特性を最適に発揮させることができ、切削工具としての寿命向上が顕著になることから好ましいことである。立方晶窒化硼系系統結体は、立方晶窒化硼が35～95体積%と残部の粒界結合相が周期律表の4a、5a、6a族元素の炭化物、窒化物、硼化物、Si、Mg、Alの窒化物、硼化物、酸化物およびこれらの相互固溶体、Co、Ni、Ti、Alの金属、合金、金属間化合物の中から選ばれた少なくとも1種からなる場合には、基材と複合硬質膜との両特性を最適に発揮させることができ、切削工具としての寿命向上が顕著になることから好ましいことである。

【0016】これらの基材に共通した問題として、基材の表面精度があり、基材の表面精度を高くすると、複合硬質膜の表面精度も高くなり、例えば、切削工具として使用した場合に摩擦抵抗が低くなって複合硬質膜表面および被削材表面の荒れが抑制されて、寿命向上効果が高くなることから好ましいことである。基材の表面精度は、JIS規格B0601に規定されている表面粗さにおける中心線平均粗さであるRaで0.1μm以下が好ましく、より好ましいのはRaが0.05μm以下からなるものである。

【0017】これらの基材表面に被覆される複合硬質膜を含む被覆層の構成は、基材に隣接して密着性を目的に被覆される下地層、この下地層に隣接して被覆される本発明における複合硬質膜でなる中間層、この中間層に隣接して被覆される外層、この外層の表面に使用前後の判別および装飾目的で被覆される最外層などを2層以上に積層する構成、具体的には、例えば基材の表面に順次被覆される被覆層が基材一下地層—複合硬質膜（中間層）—外層—最外層からなる積層の構成、基材一下地層—複合硬質膜（中間層）—外層からなる積層の構成、基材一下地層—複合硬質膜（中間層）の積層からなる構成、基材一下地層—複合硬質膜（中間層）—最外層からなる積層の構成、基材—複合硬質膜（中間層）—外層—最外層からなる積層の構成、基材—複合硬質膜（中間層）—外層からなる積層の構成、または基材—複合硬質膜（中間層）の構成、を挙げることができる。これらのうち、基材表面に直接に複合硬質膜を被覆する構成の場合には、製造時における工程の煩雑さがなく、工程時間の短縮となること、品質管理上のバラツキが少なくなることから、好ましいことである。

【0018】これらの被覆層のうち、下地層は、金属、

合金、金属間化合物または金属化合物でなり、具体的には、例えばTi、Al、Ni、Co、Wの金属、これらの相互合金、Ti—Al、Ti—Ni、Ti—Co、Al—Ni、Al—Co、Co—W、Ti—Al—Ni、Ti—Al—Coの金属間化合物、周期律表の4a、5a、6a族金属の炭化物、窒化物、炭酸化合物、窒酸化合物、これらの相互固溶体の金属化合物から選ばれた少なくとも1種の単層または多層でなる場合を挙げることができる。また、外層は、具体的には、例えば周期律表の4a、5a、6a族金属の炭化物、窒化物、炭酸化合物、窒酸化合物、これらの相互固溶体、Alの酸化合物、窒化物、酸窒化物、ダイヤモンド、硬質カーボン（ダイヤモンド状カーボンともいわれる）、立方晶窒化硼系、硬質窒化硼系、これらの2種以上の混合物の中から選ばれた少なくとも1種の単層または多層でなる場合を挙げることができる。さらに、最外層は、使用前後の判別が容易な色彩を有する被覆層、装飾の効果のある被覆層であればよく、具体的には、例えば4a、5a、6a族金属の窒化物、炭窒化物、窒酸化合物、これらの相互固溶体の中から選ばれた少なくとも1種の単層または多層でなる場合を挙げることができる。

【0019】本発明の骨子となる複合硬質膜は、被覆層自体が複合硬質膜でなる場合、別の表現をすると、基材表面に複合硬質膜のみが被覆された構成でなる場合、基材表面に上述の下地層と複合硬質膜とが被覆されている構成の場合、または前述の被覆層の構成のように中間層として被覆されている構成の場合がある。この複合硬質膜の組成成分、膜質は、具体的な例示として化学式により記載すると、(Ti, Al)N、(Ti, Al)C、(Ti, Al)(C, N)、(Ti, Al)(N, O)、(Ti, Al)(C, O)、(Ti, Al)(C, N, O)、(Ti, Al, M)N、(Ti, Al, M)C、(Ti, Al, M)(C, N)、(Ti, Al, M)(N, O)、(Ti, Al, M)(C, O)、および(Ti, Al, M)(C, N, O)の中から選ばれた少なくとも1種の単層または積層でなる場合を挙げることができる。（ただし、Mは、Ti、Alを除いた金属および半金属の元素の1種以上を表わし、特に周期律表の4a、5a、6a族元素、希土類元素、Mn元素、Mg元素、Si元素、B元素の中の少なくとも1種からなる場合が好ましい）

【0020】これらの複合硬質膜は、金属元素がTiとAlのみを含有している場合には、次の化学式で表せる(Ti_a, Al_b)(C_x, N_y, O_z)_wの複合硬質膜[ただし、aは金属元素中のTi（チタン）元素の原子比、bは金属元素中のAl（アルミニウム）元素の原子比、xは非金属元素中の炭素（C）元素の原子比、yは非金属元素中の窒素（N）元素の原子比、zは非金属元素中の酸素（O）元素の原子比、wは金属元素の合計に対する非金属元素の原子比を表し、それぞれがa + b = 1、

$0.8 \geq a \geq 0.4$ 、 $x + y + z = 1$ 、 $0.5 \geq x \geq 0$ 、 $1 \geq y \geq 0.5$ 、 $0.5 \geq z \geq 0$ 、 $1.05 \geq w \geq 0.7$ の関係にある]でなる場合には、複合硬質膜自体の強度、耐摩耗性および靱性にすぐれること、しかも歪み、欠陥が少なく耐剥離性にすぐれることから好ましいことである。

【0021】また、TiとAl以外の金属元素を含有した複合硬質膜でなる場合には、次の化学式で表せる(Ti_a, Al_b, M_{1-a-b}) (C_x, N_y, O_z)の複合硬質膜[ただし、aは金属元素中のTi(チタン)元素の原子比、bは金属元素中のAl(アルミニウム)元素の原子比、Mは周期律表の4a, 5a, 6a族元素、希土類元素、Si元素、Mn元素、Mg元素、B元素の中の少なくとも1種を表し、xは非金属元素中の炭素(C)元素の原子比、yは非金属元素中の窒素(N)元素の原子比、zは非金属元素中の酸素(O)元素の原子比、wは金属元素の合計に対する非金属元素の原子比を表し、それぞれが $0.8 \geq a \geq 0.4$ 、 $0.6 > b > 0.2$ 、 $x + y + z = 1$ 、 $0.5 \geq x \geq 0$ 、 $1 \geq y \geq 0.5$ 、 $0.5 \geq z \geq 0$ 、 $1.05 \geq w \geq 0.7$ の関係にある]でなる場合には、複合硬質膜自体の強度、耐摩耗性および靱性にすぐれること、しかも歪み、欠陥が少なく耐剥離性にすぐれることから好ましいことである。

【0022】これらの複合硬質膜は、複合硬質膜の結晶粒子の界面に金属元素からなる複合硬質膜強化物質が極微量に含有されていると、より一層複合硬質膜自体の強度、靱性がすぐれること、歪みが緩和されること、耐剥離性の向上が顕著になることから好ましいことである。このときの複合硬質膜強化物質は、基材を構成している金属元素からなる場合には、基材と複合硬質膜との整合性を高めること、密着性を高めることから好ましいことである。この複合硬質膜強化物質は、被覆層を被覆する前に、メッキ法や真空蒸着法などにより形成しておいて、これを拡散させることも可能であるが、基材を構成している金属元素を複合硬質膜中に拡散させると簡易に得られることから、好ましいことである。

【0023】これらの複合硬質膜は、複合硬質膜中のTiとAlとの金属元素の合計含有量に対するAl元素の含有量が基材表面から複合硬質膜の表面に向かって増加していること、別の表現をすると、Ti元素の含有量が複合硬質膜の表面から基材表面に向かって増加していること、いわゆる傾斜組成の複合硬質膜にすると基材と複合硬質膜との密着性がすぐれること、複合硬質膜自体の強度、靱性にすぐれて、欠陥、歪みおよび残留応力が減少すること、複合硬質膜表面の耐酸化性、耐摩耗性および耐腐食性がすぐれることから、好ましいことである。このときのAl元素およびTi元素の増加は、階段状、ノコギリの刃状にミクロ的には増減があるとしてもマクロ的には段階的に増加する場合、放物線状、直線状に連続的に増加している場合でもよいものである。

【0024】この複合硬質膜は、複合硬質膜自体の構造からすると、基材表面に対し垂直方向に柱状に成長した柱状結晶が含まれている場合には、複合硬質膜の表面からの耐圧壊強度が向上し、耐剥離性、耐微小チップング性にすぐれることから、好ましいことである。この柱状結晶を含む複合硬質膜は、具体的には、複合硬質膜全体が柱状結晶の層でなる場合、粒状結晶と柱状結晶との混在した層でなる場合、粒状結晶の層と柱状結晶の層との積層でなる場合、またはこれらの粒状結晶と柱状結晶のそれぞれの中に前述した複合硬質膜強化物質が微量含有されている場合を例示することができる。これらのうち、複合硬質膜強化物質が複合硬質膜と複合硬質膜強化物質との合計に対し、3体積%以下、好ましくは1体積%以下含有していると、複合硬質膜の表面からの垂直方向および水平方向の両方からの耐圧壊強度、耐圧縮強度にすぐれるとともに、耐摩耗性にもすぐれるというシナージ効果を発揮することができることから、好ましいことである。

【0025】これらの複合硬質膜は、前述した被覆層の構成のどの位置に存在するかにより複合硬質膜自体の構造を配慮することが好ましく、この被覆層の構成として、基材に直接複合硬質膜を被覆する場合、または基材に下地層を被覆した後、下地層に複合硬質膜を被覆し、複合硬質膜の表面が他の物質(例えば、切削工具における被削材)と接触する状態で使用される第1の構成による複合硬質膜部材と、複合硬質膜の表面に外層を被覆した場合、または複合硬質膜の表面に外層および最外層を被覆し、複合硬質膜の表面が他の物質と直接触れない状態で使用される第2の構成による複合硬質膜部材とに大別することができる。

【0026】これらのうち、第1の構成による複合硬質膜部材の場合には、複合硬質膜の表面は、JIS規格B0601に規定されている表面粗さにおける中心線平均粗さRaで $0.1 \mu m$ 以下、好ましくは $0.05 \mu m$ 以下にすると、切削工具として使用した場合に、被削材への損傷が緩和されること、切削抵抗が緩和されること、切粉の排出が容易になることから、より一層の長寿命となり、好ましいことである。また、第2の構成による複合硬質膜部材においても、外層の膜厚さ、または外層と最外層との合計膜厚さにより異なるが、複合硬質膜の表面粗さを上述のようにしておくこと、外層および最外層の表面粗さも滑らかで、平坦となり、上述と同様の効果を発揮できることから、好ましいことである。

【0027】被覆層を構成する各層の膜厚さは、用途、形状および被覆層の構成により、選択されるのであるが、上述の第1の構成による複合硬質膜部材の場合には、被覆層の主体が複合硬質膜となり、この場合には複合硬質膜の膜厚さを $1 \sim 20 \mu m$ 、主として密着性を目的として下地層を介在させる場合には、下地層の膜厚さを $0.2 \sim 2 \mu m$ とすると、被覆層自体の強度、耐摩耗

性、靱性および耐剥離性から、好ましいことである。また、上述の第2の構成による複合硬質膜部材でなる場合には、下地層の膜厚さを0.2~2 μm 、複合硬質膜の膜厚さを1~10 μm 、外層の膜厚さを1~10 μm 、最外層の膜厚さを0.5~2 μm とすると、各膜層の特性を最適に発揮させることができることから、好ましいことである。以上に詳述してきた複合硬質膜を初め、下地層、外層、および最外層は、化学量論組成でなる場合、または非化学量論組成でなる場合でもよく、実質的には非化学量論組成からなっている場合が多いものである。

【0028】以上のような形態でなる本発明の複合硬質膜部材は、各種の用途に実用できるものであり、具体的には、例えば旋削工具、フライス工具、ドリル、エンドミルに代表される切削工具、特に被削材が鋳物や鋼であり、耐衝撃性を必要とする断続切削工具や回転切削工具として、ダイス、パンチなどの型工具からスリッターなどの切断刃、裁断刃などの耐摩耗用工具として、ノズルや塗付工具などの耐腐食耐摩耗用工具として、鉱山、道路、土建などに用いられる切断工具、掘削工具、窄孔工具、破碎工具に代表される土木建設用工具として実用できるものである。これらのうち、本発明の複合硬質膜部材は、ミクロ的に温度、摩擦、熱衝撃および圧縮衝撃などが最も過酷な条件となる切削工具、特にドリル、エンドミルなどの回転切削工具、スローアウェイチップなどの切削工具として使用する場合には、複合硬質膜の特性を最適に発揮させ得ることから、好ましいことである。この複合硬質膜部材を切削工具として使用する場合には、複合硬質膜の膜厚さは、切削工具の切刃に形成される稜線部に向かって減少するように形成すると、耐剥離性、微小チップング性にすぐれることから好ましいことである。また、これらの複合硬質膜を含む被覆層の膜厚さが切削工具の切刃に形成される稜線部に向かって減少するように形成されることも、同様の効果を惹起させることになり、好ましいことである。

【0029】この本発明の複合硬質膜被覆部材は、従来から市販されているステンレス鋼、耐熱合金、高速度鋼、ダイス鋼、Ti合金、Al合金に代表される金属材料、超硬合金、サーメット、粉末ハイスに代表される焼結合金、 Al_2O_3 系統結体、 Si_3N_4 系統結体、サイアロン系統結体、 ZrO_2 系統結体、炭化珪素系統結体に代表されるセラミックス焼結体、立方晶窒化硼系統結体、ダイヤモンド系統結体に代表される超高温高压焼結体を基材とし、この基材の表面を、必要に応じて研磨し、超音波洗浄、有機溶剤洗浄などを行った後に、従来から行われているPVD法、CVD法またはプラズマCVD法により基材上に被覆層を被覆して作製することができるが、以下の方法で作製すると、プラズマ密度の向上とイオン化効率の向上が可能となること、複合硬質膜自体の気相エピタキシャル結晶成長および結晶配向が容

易となること、複合硬質膜の特性および密着性がよりすぐれることから、好ましいことである。

【0030】この複合硬質膜被覆部材を得るための製造方法として、重要な特徴について具体的に詳述すると、基材の表面は、従来から行われているブラスト処理、ショットピーニング処理、研磨処理、バレル処理の中の少なくとも1種の機械的処理と、酸性もしくはアルカリ性の電解液による電解エッチング、酸溶液、アルカリ溶液による表面腐食、または水、有機溶液による洗浄の中の少なくとも1種の化学的処理と、この機械的処理と化学的処理を同時または別々に行う処理方法とから選択される処理を行うと、基材表面の欠陥を除去できること、複合硬質膜の密着性を高め得ること、膜内歪みを抑制できること、膜内の欠陥を抑制できることから、好ましいことである。また、基材は、このような機械的処置および/または化学的処理と、低温による熱処理を付加して、上述の効果を高めることも好ましいことである。

【0031】基材の表面に複合硬質膜を被覆する場合は、スパッター法やイオンプレーティング法に代表されるPVD法により行うことが好ましく、これらのうち、マグネトロンスパッター法またはアークプラズマイオンプレーティング法により行うと、複合硬質膜の調整が容易であることから、特に好ましいことである。具体的には、例えばイオンプレーティング装置の反応容器内に基材を配置し、基材表面をボンバード処理する場合に、金属元素イオンによるボンバード処理、もしくは金属元素イオンと非金属元素イオンとの両方によるボンバード処理を施すと、上述の効果を高めることになることから、好ましいことである。特に、前述の下地層のうち、金属、合金または金属間化合物の下地層を必要とする場合は、金属元素イオンを含むイオンボンバードを施すと、下地層の形成が容易であること、基材と下地層との密着性が高くなることから、好ましいことである。

【0032】複合硬質膜の被覆条件は、反応容器の構造、プラズマの調整など装置自体の影響を重要視する必要がある、具体的には、例えば高電圧、(場合によってはパルス状高電圧と高周波を付加)の電源でイオンを加速とプラズマ発生させる装置、磁界によるプラズマの調整可能な装置を使用すること、その他、反応容器内の雰囲気圧力、温度、アーク放電電流、電圧、基材バイアス電圧、試料の配置などについて配慮する必要がある、これらのうち、従来の条件に対し、特にアーク放電電圧を高くすること、基材バイアス電圧を高くすること、試料の回転および上下動などが重要な要件である。

【0033】

【実施試験1】以上に詳述してきた本発明の実施形態について、さらに具体的な代表例として実施試験により説明する。まず、従来の配合、混合、成形、焼結の各工程を経て作製された超硬合金の基材とサーメットの基材を用いて、これらの基材表面に直接複合硬質膜を被覆した

試験について説明する。基材は、配合組成成分で示した表1により作製されたISO規格によるSNGN120408形状の超硬合金の基材1～基材5とサーメットの基材6を用い、これらの基材の上下面と外周面を270#のダイヤモンド砥石で研削加工を施し、刃先部に400#ダイヤモンド砥石により $25^{\circ} \times 0.10\text{mm}$ のホーニング加工を施し、さらに表面を湿式ブラスト処理、洗浄処理および乾燥処理を行った後、アークイオンプレーティング装置により複合硬質膜を被覆した。

【0034】処理条件は、反応容器内の各基材表面をボンバード処理した後、複合硬質膜を被覆した。ボンバード処理は、反応容器内の雰囲気：真空、基材温度：873K、アーク電流：70A、基材バイアス電圧：-600V、Arガスボンバードにより行った。複合硬質膜の被覆は、反応容器内のガス流量：200～350SCCM、蒸発源：Ti-Al合金、アーク電圧：200～300V、アーク電流：150～200A、基材温度：773～873K、基材バイアス電圧：-100～-200Vにより行い、表1に示した基材1～6のそれぞれの表面に複合硬質膜を被覆して表2に示した本発明品1～6を得た。これらのうち、反応容器内の雰囲気は、本発明品1がAr-N₂-O₂ガス組成、本発明品2がAr-N₂-COガス組成、その他本発明品3～6がAr-N₂ガス組成で行い、本発明品4の蒸発源には、Ti元素の多い合金からAl元素の多い合金に切り替えて行い、本発明品5の蒸発源には、Ti元素の多い合金-Al元素の多い合金-Ti元素の多い合金に切り替えて行った。

【0035】比較として、表1に示した基材3～6のそれぞれの表面に複合硬質膜を被覆して表2に示した比較品1～4を得た。基材表面粗さは、上述の本発明品1～6に使用の基材表面がRa=0.01～0.005μmに対し、比較品1～4に使用の基材表面がRa=0.1～0.05μmであった。比較品1～4における基材処理は、上述した本発明品の基材表面処理のうち、湿式ブラスト処理を除いて、その他はほぼ同様に処理した。また、同比較品1～4における複合硬質膜の被覆は、上述した本発明品の複合硬質膜の処理条件のうち、アーク電圧：10～50V、アーク電流：200～250A、基

材バイアス電圧：-30～-80Vとした以外は、ほぼ同様に処理した。ただし、比較品1～4の複合硬質膜処理時の蒸発源は、Ti-Al元素比が一定のものを使用した。

【0036】こうして得た本発明品1～6および比較品1～4のそれぞれの複合硬質膜について、X線回折装置、走査型電子顕微鏡、金属顕微鏡、EDS装置、ビッカース硬さ試験機および引っ掻き硬さ試験機に相当するスクラッチ試験機を用いて、複合硬質膜表面からのX線回折によるh(200)/h(111)、d(200)/d(111)、複合硬質膜表面のTiとAlの含有率、複合硬質膜表面の硬さ、スクラッチ強度を求めて、それぞれの結果を表2に示した。なお、本発明品1～6および比較品1～4の複合硬質膜厚さは、ほぼ5～7μmからなり、本発明品1～6の複合硬質膜表面の粗さは、ほぼRa=0.015～0.010μmであり、比較品1～4の複合硬質膜表面の粗さは、ほぼRa=0.15～0.10μmであった。また、本発明品1の複合硬質膜は、非金属元素中の酸素元素が1at%以下の窒酸化膜(Ti, Al)(N, O)であり、本発明品2の複合硬質膜は、非金属元素中の酸素元素が2at%以下の炭窒化膜(Ti, Al)(N, C)であり、その他の複合硬質膜は、(Ti, Al)Nで表示される窒化膜であった。

【0037】次いで、本発明品1～5および比較品1～3を用いて、以下の切削条件により湿式断続切削試験を行った。切削条件は、被削材：機械構造用炭素鋼材のS45Cに4本の溝入丸棒、切削速度：150m/min、送り：0.3mm/rev、切り込み：2.0mm、工具形状：SNGN120408、水溶性切削油により行った。湿式断続切削試験の結果は、切刃のチッピング、被覆層の剝離したとき、平均逃げ面摩耗量または境界摩耗量が0.3mmに達したときを工具寿命とし、そのときのそれぞれの切削可能時間を求めて、比較品1の寿命に対する寿命比として、表2に併記した。

【0038】

【表1】

試料番号	焼結合金の組成成分 (配合時)	重量%
基材1	97WC-3Co	
基材2	91WC-3TaC-1TiC-5Co	
基材3	88WC-2TaC-2TiC-8Co	
基材4	86.5WC-1TaC-0.5NbC-2TiC-10Co	
基材5	80WC-1Cr3C2-1VC-18Co	
基材6	26TiC-26TiN-20WC-10TaC-1Mo2C-1ZrC-8Ni-8Co	

【0039】

【表2】

試料 番号		複合硬質膜の 結晶面ピーク		複 合 硬 質 膜 の成分比、特性			切削試験 寿命比
		高さ比:h (200)/(111)	半価幅比:d (200)/(111)	Ti:Al 比	表面硬さ (HV)	スクラッチ 強度(N)	
本 発 明 品	7	7.3	1.2	58:42	2900	85	5.7
	8	7.4	1.3	53:47	2950	62	4.8
	9	7.6	1.2	55:45	2900	76	5.5
	10	7.8	1.1	54:46	2910	95	7.1
	11	8.5	1.0	50:50	3000	88	6.5
	12	7.4	0.9	51:49	2890	73	5.1
	13	7.2	0.95	57:43	2920	70	4.7
比較品5		3.3	3.0	54:46	2900	35	1.0
比較品6		3.0	2.4	53:47	2930	42	1.3

【0044】

【実施試験3】70体積% Al_2O_3 —30体積% $TiCN$ （配合組成）により作製されたセラミックス焼結体の基材7と、70体積% Al_2O_3 —30体積% SiC ウィスカー（配合組成）により作製されたセラミックス焼結体の基材8と、96体積% Si_3N_4 —1体積% MgO —1体積% Y_2O_3 —2体積% HfO_2 （配合組成）により作製されたセラミックス焼結体の基材9と、90体積% Si_3N_4 —4体積% AlN —4体積% Al_2O_3 —1体積% MgO —1体積% Y_2O_3 （配合組成）により作製されたセラミックス焼結体の基材10を用いて、実施試験1における本発明品3～6とほぼ同様に基材を処理し、各基材表面に約1 μm 膜厚さのTiを蒸着した後に、複合硬質膜を被覆し、本発明品14～17を得た。また、比較として、基材7と基材9を用いて、実施試験1における比較品1とほぼ同様に処理し、比較品7および比較品8を得た。

【0045】こうして得た本発明品14～17と比較品7及び比較品8の複合硬質膜について、実施試験1と同様にして調べて、複合硬質膜表面におけるX線回折によ

る結晶面ピーク高さ比、半価幅比、TiとAlの元素比、表面硬さ、スクラッチ強度を表5に示した。また、本発明品14と本発明品15と比較品7は、被削材：FCD600、切削速度：150m/min、切込み：1.5mm、送り：0.2mm/刃、工具形状：SNGN120408、ホーニング：0.15×—25°、乾式フライス切削試験を行った。本発明品16と本発明品17と比較品8は、被削材：FCD250、切削速度：600m/min、切込み：3.0mm、送り：0.1mm/刃、工具形状：SNGN120412、ホーニング：0.15×—25°、乾式フライス切削試験を行った。これらの乾式フライス結果は、実施試験1における切削試験と同様にし、本発明品14と本発明品15は、比較品7に対する寿命比とし、本発明品16と本発明品17は、比較品8に対する寿命比として表5に併記した。なお、本発明品14～17と比較品7及び比較品8の複合硬質膜厚さは、約5 μm であった。

【0046】

【表5】

試料 番号		複合硬質膜の 結晶面ピーク		複 合 硬 質 膜 の成分比、特性			切削試験 寿命比
		高さ比:h (200)/(111)	半価幅比:d (200)/(111)	Ti:Al 比	表面硬さ (HV)	スクラッチ 強度(N)	
本 発 明 品	1 4	6.8	0.95	60:40	2880	75	6.5
	1 5	6.3	1.05	62:38	2850	68	6.1
	1 6	12.5	1.1	48:52	3150	70	5.3
	1 7	13.4	1.2	45:55	3200	73	6.0
比較品 7		3.5	2.3	58:42	2950	31	1.0
比較品 8		3.7	2.1	46:54	3130	32	1.0

【0047】

【実施試験4】40体積% cBN —5体積% Al_2O_3 —5体積% AlN —10体積% Al —10体積% Mg —10体積% B —20体積% TiN （配合組成）により作製された超高温高压の立方晶窒化硼系統結体の基材11と、85体積% cBN —2体積% Co —5体積% Al —2体積% Mg —6体積% TiN （配合組成）により作製された超高温高压の立方晶窒化硼系統結体の基材12

と、95体積% DIA —2体積% Co —2体積% Ni —1体積% ZrC （配合組成）により作製された超高温高压の DIA 系統結体の基材13と、97体積% DIA —1体積% Co —1体積% Ni —1体積% Mg （配合組成）により作製された超高温高压の DIA 系統結体の基材14を用いて、実施試験1における本発明品3～6とほぼ同様に基材を処理し、基材11と基材12の表面に約1 μm 膜厚さのTiを蒸着し、基材13と基材14の表面に

約1 μ m厚さのNi無電解メッキを施した後に、複合硬質膜を被覆し、本発明品18～21を得た。また、比較として、基材11と基材13を用いて、実施試験1における比較品1とほぼ同様に処理し、比較品9および比較品10を得た。

【0048】こうして得た本発明品18～21と比較品9及び比較品10の複合硬質膜について、実施試験1と同様にして調べて、複合硬質膜表面におけるX線回折による結晶面ピーク高さ比、半価幅比、TiとAlの元素比、表面硬さ、スクラッチ強度を表6に示した。また、本発明品18と本発明品19と比較品9は、被削材：S

CM415（硬さ：約HRC61）、切削速度：150m/min、切込み：0.5mm、送り：0.1mm/rev、工具形状：Tんま160408、ホーニング：0.15 \times 25 $^{\circ}$ 、外周連続乾式旋削試験を行い、その結果を表6に併記した。この外周連続乾式旋削試験の評価は、実施試験1における切削試験と同様に行い、比較品9に対する寿命比として表した。なお、本発明品18～21と比較品9及び比較品10の複合硬質膜厚さは、約5 μ mであった。

【0049】

【表6】

試料 番号		複合硬質膜の 結晶面ピーク		複 合 硬 質 膜 の成分比、特性			切削試験 寿命比
		高さ比：h (200)/(111)	半価幅比：d (200)/(111)	Ti：Al 比	表面硬さ (HV)	スクラッチ 強度(N)	
本 発 明 品	18	19.5	1.1	58：42	2960	85	8.2
	19	18.4	1.0	59：41	2930	83	8.8
	20	8.5	1.05	55：45	2950	76	なし
	21	8.9	0.95	54：46	2970	74	なし
比較品9		3.1	2.0	55：45	2910	23	1.0
比較品10		3.5	1.7	52：48	2940	25	なし

【0050】

【発明の効果】本発明の複合硬質膜被覆部材は、気相法エビタキシャルによる結晶成長と結晶配向による複合硬質膜が被覆されていること、複合硬質膜自体の歪み、欠陥が抑制されていること、微細結晶であること、場合によっては柱状晶結晶および／または微量の金属などの複合硬質膜強化物質が含まれた複合硬質膜であることから、従来の複合硬質膜被覆部材または本発明から外れた複合硬質膜被覆部材に対比して、基材と複合硬質膜、下地層と複合硬質膜、複合硬質膜と外層など、複合硬質膜

に隣接する物質に対し、密着性および耐剥離性が非常にすぐれること、複合硬質膜自体の高靱性、高強度、耐熱性、耐熱衝撃性、耐酸化性および耐摩耗性がすぐれていること、その結果として例えば切削工具として使用した場合に、切削工具として重要視される高靱性、耐摩耗性、耐熱衝撃性、耐欠損性、耐酸化性および耐溶着性が顕著に向上し、長寿命化が達成されること、切削加工における高効率化が達成されること、バラツキが小さく安定しているという顕著な効果がある。

【手続補正書】

【提出日】平成12年12月8日（2000.12.8）

【手続補正1】

【補正対象書類名】明細書

【補正対象項目名】0032

【補正方法】変更

【補正内容】

【0032】複合硬質膜の被覆条件は、反応容器の構造、プラズマの調整など装置自体の影響を重要視する必要がある、具体的には、例えば高電圧（場合によっては

パルス状高電圧と高周波を付加）の電源でイオンを加速し、プラズマを発生させる装置、さらに磁界によるプラズマの調整可能とした装置を使用すること、その他反応容器内の雰囲気圧力、温度、アーク放電電流、電圧、基材バイアス電圧、試料の配置などについて配慮する必要があり、これらのうち、従来の条件に対し、特にアーク放電電圧を高くすること、基材バイアス電圧を高くすること、試料の回転および上下動などが重要な要件である。

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